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COMPATIBILITY TESTING OF SPACECRAFT MATERIALS
AND SPACE-STORABLE LIQUID PROPELLANTS

FINAL REPORT

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NET PROPULSION LABORATORY

COMPATIBILITY TESTING OF SPACECRAFT MATERIALS
AND SPACE-STORABLE LIQUID PROPELLANTS

FINAL REPORT

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ABSTRACT

Compatibility data for aluminum alloy 2219-T87 and titanium alloy Ti-6Al-4V were obtained while these alloys were exposed to both liquid and vapor fluorine and FLOX at $-320^{\circ}\text{F} \pm 10^{\circ}\text{F}$. These data were obtained using a new low cost compatibility method which incorporates totally sealed containers and double dogbone test specimens and propellants in the simultaneous exposure to vapor and liquid phases. The compatibility investigation covered a storage period in excess of one year. Pitting was more severe in the 2219-T87 aluminum alloy than in the Ti-6Al-4V titanium alloy for both fluorine and FLOX exposure. The degree of chemical attack is more severe in the presence of FLOX than in fluorine and the more severe in the liquid phase exposure than in the vapor phase. The mechanical properties of the two alloys were not affected by storage in either of the two propellants.

FOREWORD

This report was prepared by TRW Systems Group, Redondo Beach, California, and represents the program efforts between 29 June 1972 and 15 January 1974. The work was performed under JPL Contract No. 953486 for the purpose of determining the compatibility of spacecraft materials and space-storable liquid propellants. The program was originated and managed by the Jet Propulsion Laboratory under the technical direction of O. F. Keller initially and L. Toth in the latter program stages. The TRW Systems Program Manager is Mr. J. R. Denson with overall program management provided by Dr. E. A. Burns, Manager, Applied Chemistry Department

Substantial support and assistance was provided by Mr. W. B. Turner.

CONTENTS

	<u>Page</u>
1.0 INTRODUCTION AND SUMMARY	1
1.1 Objectives	1
1.2 Specific Tasks	2
2.0 TASK I - MODIFICATION OF PREVIOUS PLAN.	5
3.0 TASK II - INTRODUCTION TO STORAGE OF THE PROPELLANTS AND MATERIALS OF CONSTRUCTION	11
4.0 TASK III - POST TEST CHARACTERIZATION	13
4.1 Establishing Method for Propellant and Specimen Removal.	13
4.2 Phase I - Post Test Characterization	13
4.2.1 Visual and Microscopic Examination.	16
4.2.2 Electron Microprobe Analysis.	19
4.2.3 Residual Mechanical Properties.	19
4.2.4 Metallographic Examination.	25
4.3 Phase II - Post Test Characterization.	26
4.3.1 Characterization of Propellant and Residues . .	29
4.3.2 Visual and Microscopic Characterization and Tensile Testing	32
4.3.3 Metallographic Characterization	32
4.4 Phase III and Phase IV - Post Test Characterization. .	38
5.0 CONCLUSIONS AND RECOMMENDATIONS	47
APPENDIX A	A-1
APPENDIX B	B-1
APPENDIX C	C-1
APPENDIX D	D-1
APPENDIX E	E-1
APPENDIX F	F-1
APPENDIX G	G-1
APPENDIX H	H-1
APPENDIX I	I-1

LIST OF TABLES

		<u>Page</u>
I	Specimens in Test	3
II	Composition of Metal Samples.	11
III	Boiling Points and Melting Points of Potential Materials in Capsules	14
IV	Tensile Test Results of Propellant Exposed Specimens. .	25
V	Weight of Specimens Before and After.	25
VI	Pit Characterization.	26
VII	Composition of Propellants and Residue at Storage Temperature	30
VIII	Composition Specimen Surface Washings	31
IX	Tensile Test Results.	34
X	Quantitative Analysis of Pitting.	38

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ILLUSTRATIONS

	<u>Page</u>
Figure 1. Sample in Sealed Capsule	7
Figure 2. Capsule in "Cryofunnel" Being Loaded with Propellant	8
Figure 3. Liquid Nitrogen Filled Specimen Capsule Storage Tank	8
Figure 4. Storage Area	9
Figure 5. Tensile Specimens After Propellant Immersion . . .	16
Figure 6. Interface of Liquid and Vapor in Specimen 7234 . .	17
Figure 7. Interface Liquid and Vapor in Specimen 7234. . . .	17
Figure 8. Specimen 7234 Showing White Deposits in Streaks. .	18
Figure 9. Specimen 7234 Showing White Crystalline Deposits .	18
Figure 10. Mapping of Surface Appearance of Double Dogbone Specimen 7234 After Exposure	20
Figure 11. Surface Microstructure of Vapor Side of Ti-6Al-4V Specimen 7234.	21
Figure 12. Surface Microstructure of Liquid Side of Specimen 7234 Showing Pits.	21
Figure 13. SEM Photo of Surface of Specimen 7234 Vapor Exposure	22
Figure 14. SEM Photo of Surface of Specimen 7234 Liquid Exposure	23
Figure 15. Tensile Tested Ti-6Al-4V Specimens	24
Figure 16. Cross Section of Vapor Exposed Part of Specimen 7234 Showing Reacted Surface Profile	27
Figure 17. Cross Section of Liquid Exposed Part of Specimen 7234 Showing Reacted Surface Profile	28
Figure 18. Mapping of Surface Appearance of Double Dogbone Specimens 7208, 7220, 7244 and 7210.	33
Figure 19. Double Dogbone Specimens After Exposure.	35
Figure 20. Tensile Tested 2219-T87 Aluminum Specimens Showing Type of Fracture	36
Figure 21. Tensile Tested Ti-6Al-4V Specimen Showing Type of Fracture	37
Figure 22. SEM Photos of 2218-T87 Aluminum Specimen 7220 After Exposure to FLOX	39
Figure 23. SEM Photos of 2219-T87 Aluminum Specimen 7208 After Exposure to Fluorine	40

ILLUSTRATIONS (CONTINUED)

	<u>Page</u>
Figure 24 SEM Photos of Ti-6Al-4V Specimen 7210 After Exposure to Fluorine	41
Figure 25 SEM Photos of Ti-6Al-4V Specimen 7244 Surface After Exposure to FLOX	42
Figure 26 Cross Section of 2219-T87 Aluminum Alloy Specimen 7220 Showing Reacted Surface Profile	43
Figure 27 Cross Section of 2219-T87 Aluminum Alloy Specimen 7208 Showing Reacted Surface Profile	44
Figure 28 Cross Section of Ti-6Al-4V Specimen 7244 Showing Reacted Surface Profile.	45
Figure 29 Cross Section of Ti-6Al-4V Specimen 7210 Showing Reacted Surface Profile.	46
Figure C-1 Double Dogbone Test Specimen	C-2
Figure C-2 Stressed Test Specimen	C-4
Figure C-3 Stressing of Bent-Beam Stress Corrosion Specimen	C-5
Figure D-1 Sealed Compatibility Test Capsule.	D-2
Figure E-1 Propellant Handling Manifold	E-2
Figure E-2 Canister Modification.	E-5
Figure F-1 Propellant Handling Manifold	F-2
Figure G-1 Conditioning Flasks and Controls	G-2
Figure H-1 Canister Modification.	H-2
Figure H-2 Capsule Opening Device	H-3
Figure H-3 Propellant Handling Material	H-4

1. INTRODUCTION AND SUMMARY

This document is the final program report describing work performed by TRW Systems for the Jet Propulsion Laboratory under Contract 953486 during the period July 1, 1972 through March 5, 1974. The principal objective of this program was to obtain vital long term propellant/material of construction compatibility information for design of a liquid propulsion system using liquid fluorine or FLOX (88/12 F_2/O_2 mixtures). To accomplish this primary objective a new low cost method for conducting compatibility measurements at cryogenic temperatures was established through modification of a previously documented plan, evaluated and shown to be acceptable for obtaining this critically needed information. Sensitive post test characterization methods were employed which permit observations of subtle changes in the corrosion resistance of the two test materials, aluminum 2219-T87 alloy and titanium 6Al-4V alloy. Because of the high sensitivity of the post exposure characterization methodology, clear-cut distinction regarding the compatibility of these two materials in the presence of liquid fluorine and FLOX was obtained over 10- and 12-month storage periods. However, extended storage periods are recommended to ascertain whether these changes continue as a function of time to such an extent that they could seriously affect the reliability of the materials when used as hardware in advanced missions to outer planets which will require contact with the propellants for periods as long as five to ten years.

1.1 OBJECTIVES

The objectives of this program were to obtain critical long-term propellant/material of construction compatibility information for design of a liquid propulsion system with liquid fluorine and FLOX. Hardware commitments to advanced missions to outer planets cannot be made without identification of acceptably inert materials having the high performance and reliability after long periods of storage in the mission environment while in contact with either liquid fluorine or FLOX at a temperature of $-320^{\circ}F \pm 10^{\circ}F$.

The compatibility understanding and planning activities conducted by TRW for JPL under Contract No. 952964 are directly applicable to obtaining the required fluorine and FLOX material compatibility information. The

plan generated in that program for material compatibility in oxygen difluoride (OF_2) and diborane (B_2H_6) liquid propellant is described in Report No. 16681-6009-RO-00, dated April 1971 and referred to in this report as the Previous Plan. This Previous Plan serves as the basis for modification to permit determination of compatibility of materials with the liquid propellants fluorine and FLOX. It is a second objective of this program to evaluate the adequacy of the revised material compatibility plan for obtaining meaningful information.

1.2 SPECIFIC TASKS

The objectives identified above were accomplished through implementation of the activities described below as specific project tasks:

- TASK I - Modification of Previous Plan

The procedures from the Previous Plan were modified where necessary for substitution of the two propellants fluorine and FLOX for OF_2 and B_2H_6 .

- TASK II - Introduction to Storage of the Propellants with the Materials of Construction

This activity consists of placing into storage during the week of November 13, 1972 the propellants/materials of construction and appropriate reference materials.

- TASK III - Compatibility Tests

Twenty-four (24) double dogbone test specimens as listed in Table I were placed in storage. These specimens were removed at three time periods for post-test characterization.

- TASK IV - Post-Test Characterization

Post-test characterization of the propellants, materials and their interaction products began on test termination. The testing was done in four phases. The first phase consisted of examination after storage to 24 July 1973 of the propellant and test specimens as follows:

- One (1) capsule containing titanium and liquid fluorine
- One (1) capsule containing titanium and liquid FLOX

TABLE I
SPECIMENS IN TEST

Specimen No.	Sample Material	Liquid Propellant
7202	A12219-T87	Fluorine
7204	A12219-T87	Fluorine
7206	A12219-T87	Fluorine
7208	A12219-T87	Fluorine
7220	A12219-T87	FLOX
7222	A12219-T87	FLOX
7224	A12219-T87	FLOX
7226	A12219-T87	FLOX
7238	A12219-T87	FLOX
7218	A12219-T87	Reference, Vacuum Sealed
7240	A12219-T87	Reference, Vacuum Sealed
7242	A12219-T87	Reference, Vacuum Sealed
7210	6A1-4V Titanium	Fluorine
7212	6A1-4V Titanium	Fluorine
7214	6A1-4V Titanium	Fluorine
7216	6A1-4V Titanium	Fluorine
7228	6A1-4V Titanium	FLOX
7230	6A1-4V Titanium	FLOX
7234	6A1-4V Titanium	FLOX
7236	6A1-4V Titanium	FLOX
7244	6A1-4V Titanium	FLOX
7232	6A1-4V Titanium	Reference, Vacuum Sealed
7246	6A1-4V Titanium	Reference, Vacuum Sealed
7248	6A1-4V Titanium	Reference, Vacuum Sealed
Control	None	Fluorine
Control	None	Fluorine
Control	None	Fluorine
Control	None	FLOX
Control	None	FLOX
Control	None	FLOX

Phase II consisted after storage to 26 August 1973 of characterization of the propellant and test specimens and the interaction products from the following units:

- One (1) capsule containing aluminum and liquid fluorine
- One (1) capsule containing titanium and liquid fluorine
- One (1) capsule containing aluminum and FLOX, and
- One (1) capsule containing titanium and FLOX

Phase III consists of the post test characterization of the propellant and test specimens and the interaction products of propellants with the specimens and/or capsule for the following units after storage for 60 weeks:

- One (1) capsule containing aluminum and liquid fluorine
- Two (2) capsules containing titanium and liquid fluorine
- One (1) capsule containing aluminum and FLOX
- One (1) capsule containing titanium and FLOX
- One (1) capsule containing liquid fluorine
- One (1) capsule containing liquid FLOX

Phase IV consists of the post test characterizations of propellant and test specimens and the interaction products of propellants with the specimens and/or capsules of the remaining units after a total storage time of approximately 70 weeks.

The results of all investigations conducted as part of Phase III and Phase IV will be submitted as an appendix to this report.

2. TASK I - MODIFICATION OF PREVIOUS PLAN

The number of variables and the number of levels for each variable to be studied, e.g., temperature, materials, material combinations, propellant composition, unstressed and degree of stress, testing durations and replication and references, provides a compatibility program with a large number of specimens. For a long term program, the cost of the program becomes very high, particularly when cryogenic storage requirements and post test chemical and metallographic characterization are considered. Superimposed on the costs for these tests are special safety requirements because of the hazardous nature of liquid fluorine and FLOX. For these reasons the requirements for compatibility measurements at -320°F using FLOX and liquid fluorine were reviewed and assessed and a new approach for these measurements was generated which meet the criteria of providing:

- Meaningful technical information,
- Relatively low cost operation, and
- Safe handling over extended storage periods.

Previously TRW devised a plan for conducting a material compatibility program using oxygen difluoride OF_2 and diborane B_2H_6 liquid propellants.^{1,2} This plan incorporated glass containers which could be readily sealed without the introduction of contaminants, double dogbone metal specimens and storage in a cryoflask canister assembly. Because this plan had never been tested experimentally, a key requirement for its successful implementation was the requirement that no chemical attack of the container walls occurred. As described in Section 4, Post Test Characterization, no significant etching of the glass containers was observed throughout these tests and no failures resulted during the execution of the normal operating procedures.

¹TRW Document No. 16681-6009-RO-00, A Plan for a Material Compatibility Program for Oxygen Difluoride (OF_2) and Diborane (B_2H_6) Liquid Propellants, Final Report dated April 1971

²Compatibility Testing of Spacecraft Materials and Space Storable Liquid Propellants by J. R. Denson and E. A. Burns. Paper given at the Fourth Working Group on Propellant Characterization. San Diego, California, September 12, 1973.

During the course of the Phase I post test characterization, a change in the propellant sampling procedure was implemented to ensure safe acquisition of propellant.

The procedure for performing the tasks in this program are presented in Appendices A through H. These appendices are modifications of those given in the Previous Plan. The procedures were modified to incorporate the modifications as follows:

- A change in storage temperature from -320°F to $-320^{\circ}\text{F} \pm 10^{\circ}\text{F}$
- The substitution of the two propellants fluorine and FLOX for OF_2 and B_2H_6
- A change in the specimen and capsule cleaning procedure to comply with JPL Process Specification FS506300, Rev. A dated 26 October 1971, "General Cleaning Requirement for Liquid Fluorine and FLOX Propulsion Subsystem and Support Equipment, Detail Specification For"
- Modification of the specimen drawing to comply with JPL Drawing 10056227, Rev. B, "Metallic Specimen, Double Dogbone"
- Modification of the propellant filling and withdrawal operations to accommodate for the propellants fluorine and FLOX

With the exception of changes in the propellant filling and withdrawal operations, the required changes to the operating procedures for OF_2 compatibility testing were minor, and in most cases were only required because of the lower test temperature, -320°F . Because fluorine has a vapor pressure of about 0.4 atm at -320°F and oxygen has a vapor pressure of about 0.1 atm at -320°F , the propellants cannot be loaded by vacuum transfer methods at liquid nitrogen temperatures (-320°F).

Liquid helium, neon and hydrogen baths will, however, freeze fluorine and FLOX and both have a very low vapor pressure when frozen, 0.1 torr for fluorine and about 2 microns for oxygen. For safety reasons, liquid hydrogen baths were not considered for use. Neon has a much greater heat of vaporization than helium, approximately 40 times that of helium on an equivalent liquid volume basis. However, neon costs about 20 times as much as

helium. After a review of the handling techniques with cryogenic equipment engineers, it was determined that most of the cryogenic liquid would be lost in boil-off in use of the "Cryofunnel"* and it became evident that adequate advantage could not be taken of the greater heat of vaporization for neon. Therefore, a decision was made to use helium as the cryogen for loading and sealing operations.

Appendix C "Procedure for Preparation of Test Specimens" includes instructions for preparation of non-metallic, stressed and dissimilar metal specimens. This type specimen was not used in this program, but instructions for their preparation is included for use in future investigations which may require such specimens. Pictures showing various stages of the operations are shown in Figures 1 - 4.

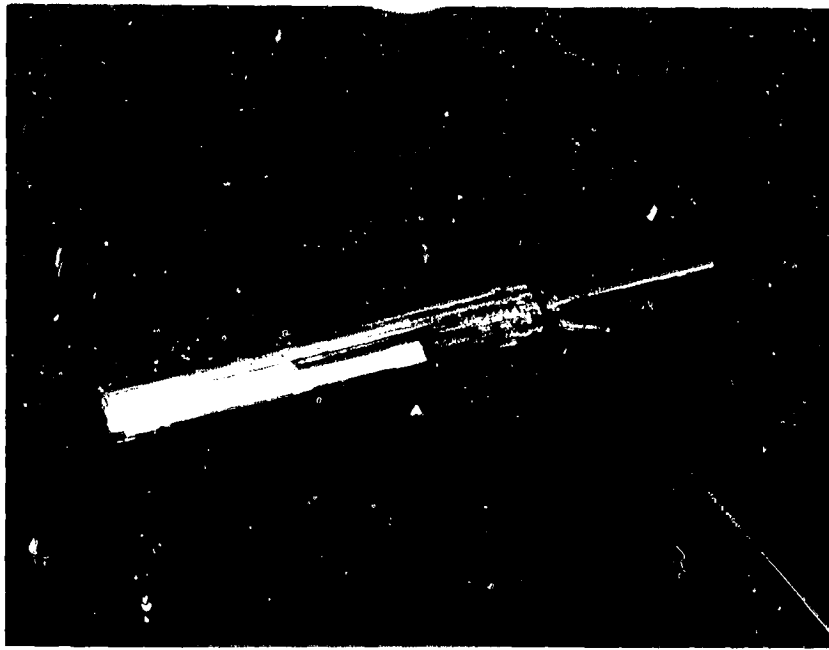


Figure 1. Sample in Sealed Capsule

*The "Cryofunnel" is a simple dewar attached to a liquid helium flask for handling liquid helium (see Figure 2). Cryofunnels are marketed by Cryogenic Service Corporation.

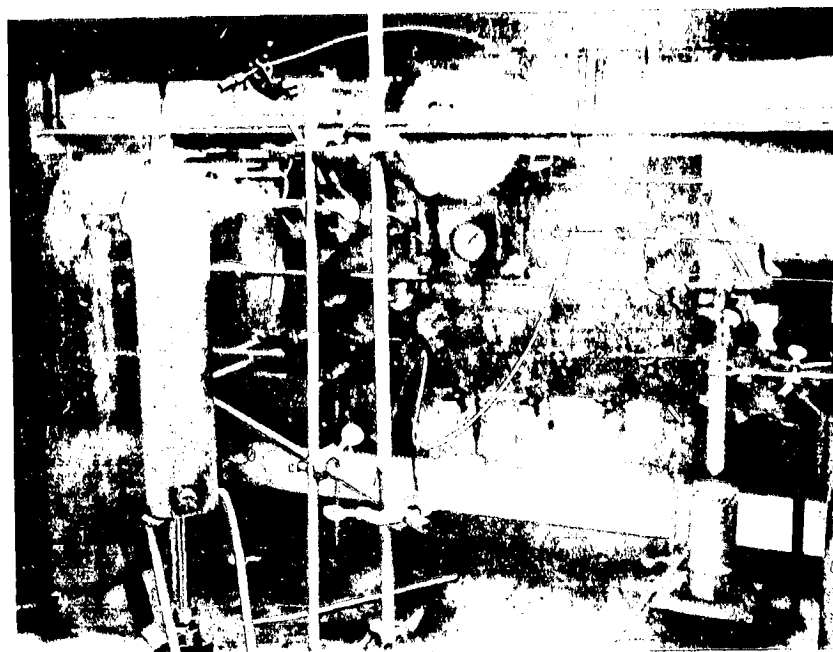


Figure 2. Capsule in "Cryofunnel" Being Loaded with Propellant



Figure 3. Liquid Nitrogen Filled Specimen Capsule Storage Flask



Figure 4. Storage Bunker

3. TASK II - INTRODUCTION TO STORAGE OF THE PROPELLANTS AND MATERIALS OF CONSTRUCTION

Before initiating the test, the necessary steps outlined in the Appendices B and C were performed. FLOX was prepared according to the instructions given in Appendix B. The double dogbone standard specimen was manufactured and cleaned according to the instructions given in Appendix C. The certified analyses for the aluminum and titanium specimens were performed by Durker Testing Laboratories, Gardena, California. The results of these analyses are given in Table II. The capsules were manufactured ac-

TABLE II
COMPOSITION OF METAL SAMPLES

Element	% (w/w) Composition	
	2219-T87 Aluminum	6Al-4V Titanium
Aluminum	Remainder	6.23
Copper	6.18	
Magnesium	<0.01	
Manganese	0.31	
Iron	0.14	0.12
Silicon	0.16	
Vanadium	0.09	4.26
Zirconium	0.14	
Chromium	<0.01	
Zinc	0.04	
Titanium	0.02	Remainder
Nickel	<0.01	

cording to Appendix D. Analysis of the fluorine from Matheson Gas Products after removal of hydrogen fluoride with a sodium fluoride trap, indicated the composition to be $88\% \pm 0.5\%$ fluorine and $12\% \pm 0.5\%$ oxygen. After sealing the specimens in the capsules according to Appendix D, the capsules were filled with about 5 ml of liquid propellant according to Appendix E and placed in storage according to Appendix G. The ullage volume in each capsule was about 12 ml.

All operations associated with manufacture of capsules and specimens, cleaning of capsules and specimens, preparation of FLOX, sealing specimens in capsules, and introduction of propellant into capsules went smoothly with one exception. During the introduction of fluorine into the capsules containing titanium sample No. 7246 and aluminum sample No. 7240, the filling ports on the capsules broke. The fluorine was allowed to distill at room temperature, the capsules were dried at 40°C overnight, and the capsules were sealed. These two specimens were held as reference specimens but the aluminum sample 7240 has a stain which makes its use as a reference questionable.

Photographs were taken of various steps in the preparation of specimens for storage and the storage operation. The photographs are shown as Figures 1 through 4.

4. TASK III - POST TEST CHARACTERIZATION

4.1 ESTABLISHING METHOD FOR PROPELLANT AND SPECIMEN REMOVAL

In the entire set of operating procedures given in the Appendices, the procedure for opening the capsules and removing the specimens in a safe manner was of the greatest concern. For this reason the two Phase I specimens were selected for evaluation of the proposed procedure for opening the capsule and examination of propellant and test specimens. Specifically, the specimens selected were 6Al-4V titanium alloy specimen 7214, which was stored in fluorine, and specimen 7236 of the same alloy, which was stored in FLOX. These two specimens were in storage from 13 November 1972 to 26 September 1973 (ten months, 13 days).

The capsule containing specimen 7214 was not opened using the revised procedure described in Appendix H, but instead the propellant was allowed to distill into a flowing stream of helium. The propellant was allowed to warm very slowly by lowering slowly a liquid nitrogen filled dewar flask in which the capsule was immersed. About 80% of the fluorine distilled from the flask and then an explosion occurred. The titanium sample was not damaged and was recovered. It was washed with distilled water, dried, and placed in a polyethylene bag. The procedure was then revised to permit vacuum distillation of the propellant from the capsule while the capsule remained 80% immersed in a liquid nitrogen bath. The revised procedure is presented in Appendix H. The FLOX propellant was removed safely from specimen 7236 using this procedure. No other incidents were encountered in handling and test termination using the revised procedure.

4.2 PHASE I - POST TEST CHARACTERIZATION

Phase I consisted of the examination after a storage period of ten months of the propellant and test specimens as follows:

- One (1) capsule containing titanium and liquid fluorine
- One (1) capsule containing titanium and liquid FLOX

The purpose of Phase I was to evaluate and assess the adequacy of the operational procedures, make appropriate modifications and obtain post test characterization information.

The propellants in the vapor phase of the capsule containing specimen 7214 were analyzed by infrared spectrometry and gas chromatography by the techniques described in MIL-P-27413, "Specification for Chlorine Pentafluoride" with the exception that a 1-foot x 0.25-inch column packed with sodium chloride was placed before the usual column.

The vapor in the capsule containing specimen 7216 and fluorine was analyzed before the reported explosion with the results indicating no HF, CF_4 or CO_2 present, but 2.4 mol percent SiF_4 was present. The presence of silicon tetrafluoride in the ullage volume of the compatibility capsule was a surprise because at the storage temperature of -195.8°C (-320°F) silicon tetrafluoride exists as a solid. Undoubtedly, this material was generated during transfer of gas to the infrared gas cell and hence, was not a product of the compatibility test. Table III lists boiling points and melting points of compounds that could be expected to be in the compatibility capsules through either direct transfer (e.g., the propellants) or through inadvertent contamination or reaction with contaminants on the test specimens and capsules.

TABLE III
BOILING POINTS AND MELTING POINTS
OF POTENTIAL MATERIALS IN CAPSULES

	Boiling Point $^\circ\text{C}$	Melting Point $^\circ\text{C}$
Nitrogen	-195.8	-209.9
Carbon Monoxide	-191.5	-199
Fluorine	-188	-223
Oxygen	-183.0	-218.4
Carbon tetrafluoride	-128	-184
Silicon tetrafluoride	-86	-90.2
Carbon Dioxide	-78.5*	*
Hydrogen Fluoride	+19.5	-83.1
Sulfur Hexafluoride	+63.8	-50.5

*Sublimes

A calculation was made to determine the maximum extent of corrosion of the glass capsule if the observed silicon tetrafluoride in the gas phase was formed by the attack of the compatibility capsule. For this calculation it was assumed the gas phase had a temperature of -86°C . A concentration of 2.4 mole percent at a total pressure of 319 torr at 187°K corresponds to a weight of silicon tetrafluoride of $700\mu\text{g}$. This amount corresponds to a consumption of $400\mu\text{g}$ silicon dioxide. This quantity would clearly be negligible with respect to the total mass of the glass capsule, but does indicate that the propellants may react with a "warm" capsule. Post test examination of the glass compatibility container indicated there was no etching of the surfaces. This finding confirms the projected viability of this procedure as discussed in the previous compatibility plan (References 1 and 2).

The propellant in the second capsule containing specimen 7236 was not analyzed during the initial distillation because of the concern of the explosion. The FLOX was removed from the capsule by vacuum distillation while the capsule was 80% immersed in liquid nitrogen. The FLOX was passed through a soda lime and then a charcoal scrubber before entering a vacuum pump. The soda lime scrubber became warm (130°F) while no heating was noted in the charcoal scrubber. The last propellant to be removed from the capsule was examined by infrared spectroscopy, but no impurities were discerned.

As previously indicated, specimen 7214 is a double dogbone Ti-6Al-4V sheet specimen which has been exposed to part liquid and part gaseous fluorine environment. The glass capsule exploded during the course of the experiment and the specimen was cleaned in distilled water, air dried, and placed in a polyethylene bag. This specimen had also been exposed to ambient air environment for about three weeks prior to metallographic characterization. Therefore, the surface conditions at the time of characterization may not be truly representative of the propellant effect. For this reason this specimen was subjected only to limited physical and microscopic analyses.

Specimen 7234 was handled in accordance with the revised plan; that is, upon completion of the exposure, the FLOX propellant was vacuum-distilled and the capsule was back-filled with helium and fusion-sealed. This specimen was subjected to a more thorough examination.

4.2.1 Visual and Microscopic Examination

Visual examination was performed on both specimens 7214 (fluorine exposure) and 7234 (FLOX exposure) at magnifications up to 40X using a stereo microscope and up to 800X using a metallograph. Specimen 7214 showed uniform surface texture and no sign of chemical attack at all magnifications, although slight discoloration was observed in spots on the surface. It is concluded that specimen 7214 was unaffected in fluorine for the duration of the test and that further microscopic and chemical analysis was not necessary. A macrophotograph of the specimens is shown in Figure 5. In specimen 7234, the liquid-vapor interface on the specimen surface is very distinct

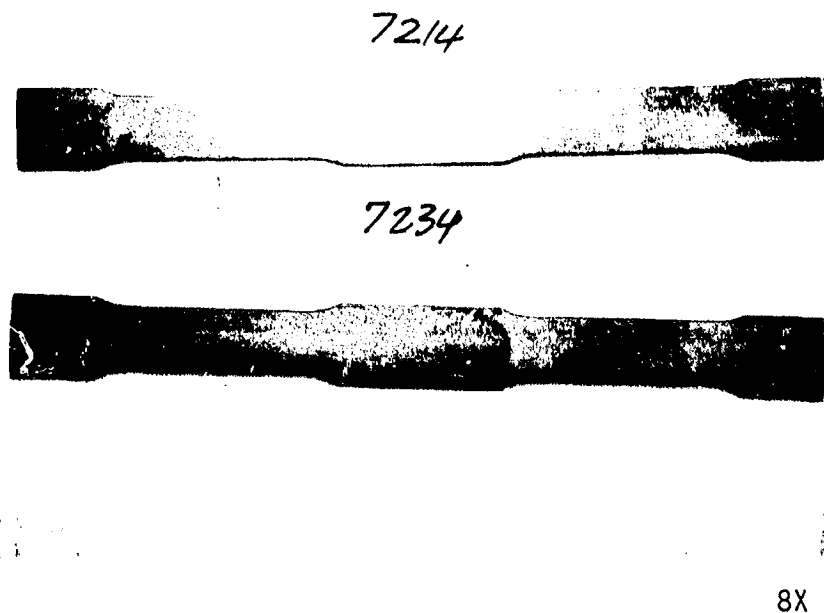


Figure 5. Tensile Specimens After Propellant Immersion
 7214 - Liquid Fluorine
 7234 - FLOX

(Figures 5, 6 and 7). The liquid-exposed area showed not only dark discoloration, but also streaks of pits and very fine white deposits (Figures 8 and 9).



8X

Figure 6. Interface of Liquid (Dark Area) and Vapor (Light Area) in Specimen 7234



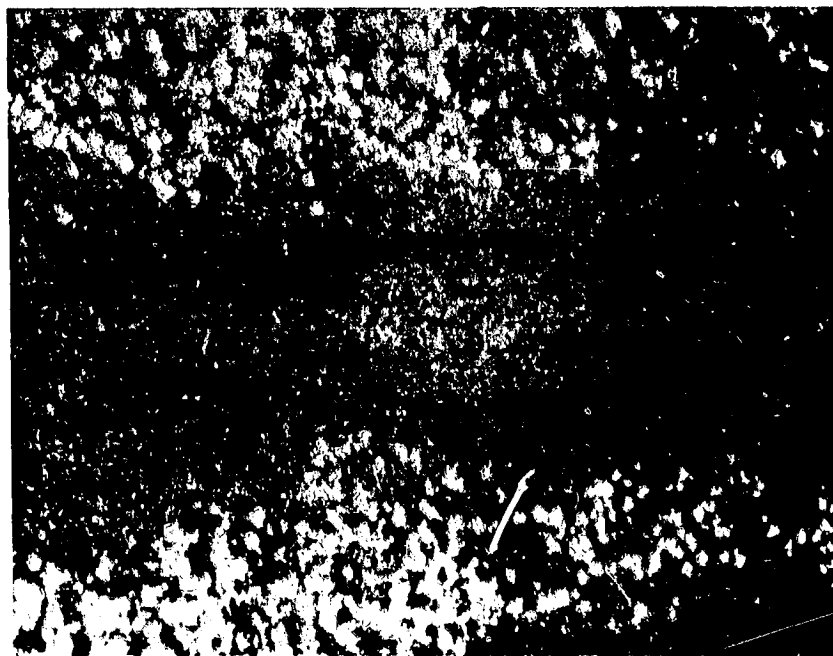
(32X)

Figure 7. Interface Liquid (Dark Area) and Vapor (Light Area) in Specimen 7234



(8X)

Figure 8. Specimen 7234 Showing White Deposits
in Streaks



(32X)

Figure 9. Specimen 7234 Showing White Crystalline Deposits

The vapor-exposed position showed no evidence of macroscopic pits. A mapping of the surface condition as observed at magnifications up to 40X is presented in Figure 10. Typical surface appearance as seen at higher magnifications in the metallograph is shown in Figures 11 and 12. The spots shown in Figure 11 were not evident at low magnifications. The surface conditions of the vapor- and liquid-exposed areas were then further examined in the scanning electron microscope (SEM) to provide greater resolution. The results are shown in Figure 13 and 14 for vapor and liquid exposed areas, respectively. It is evident from these photos that the vapor-exposed surface (Figure 13) also contains microscopic pits and is considerably smoother than the liquid-exposed area (Figure 14). The liquid-exposed surface is rough and contains numerous microscopic as well as macroscopic pits.

4.2.2 Electron Microprobe Analysis

A sample taken from the vapor-exposed area and also from the liquid-exposed area was subjected to electron microprobe analyses. Spectral scans were made, followed by a search specifically for oxygen and fluorine on the surface. Results showed no foreign elements other than the alloying elements in Ti-6Al-4V and oxygen. Fluorine was not detected. It is, therefore, concluded that the reaction products on the surface are oxides of the metallic elements. The chemical composition of the minute white particles observed microscopically was not determined.

4.2.3 Residual Mechanical Properties

The double dogbone specimens 7214 and 7234 were each cut into two tensile test specimens: vapor-exposed and liquid-exposed. The specimens were tested in accordance with the ASTM method in an Instron test machine. Results are summarized in Table IV. The yield strength, ultimate tensile strength and percent elongation of all specimens are about equal and typical for unexposed annealed Ti-6Al-4V sheet material. Therefore, it is concluded that the tensile properties were not affected by exposure to FLOX for this duration. The tensile break of all specimens as shown in Figure 15 showed typical 45° ductile fracture.

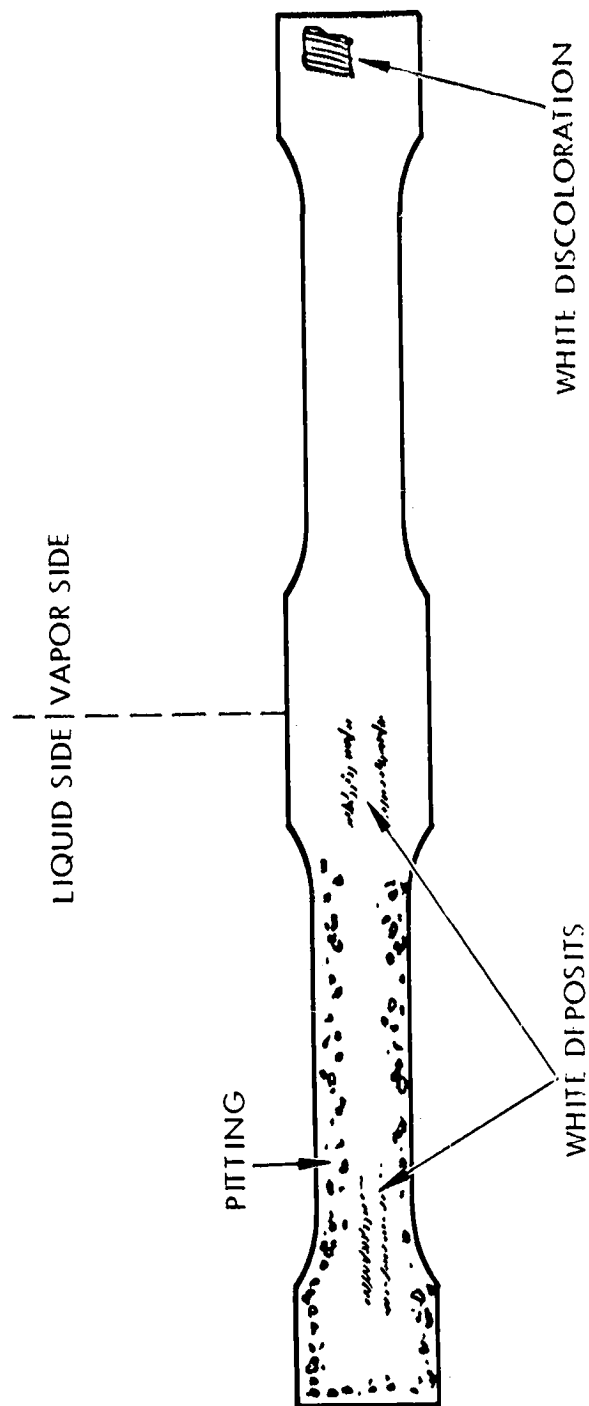
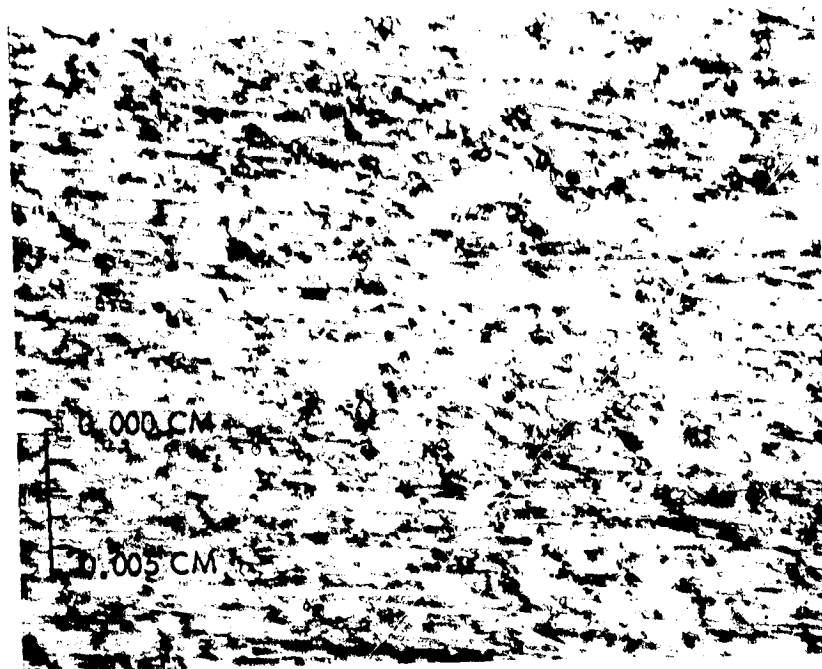


Figure 10. Mapping of Surface Appearance of Double Dogbone Specimen 7234 After Exposure



(400X)

Figure 11. Surface Microstructure of Vapor Side of Ti-6Al-4V Specimen 7234



(400X)

Figure 12. Surface Microstructure of Liquid Side of Specimen 7234 Showing Pits (Dark Spots)

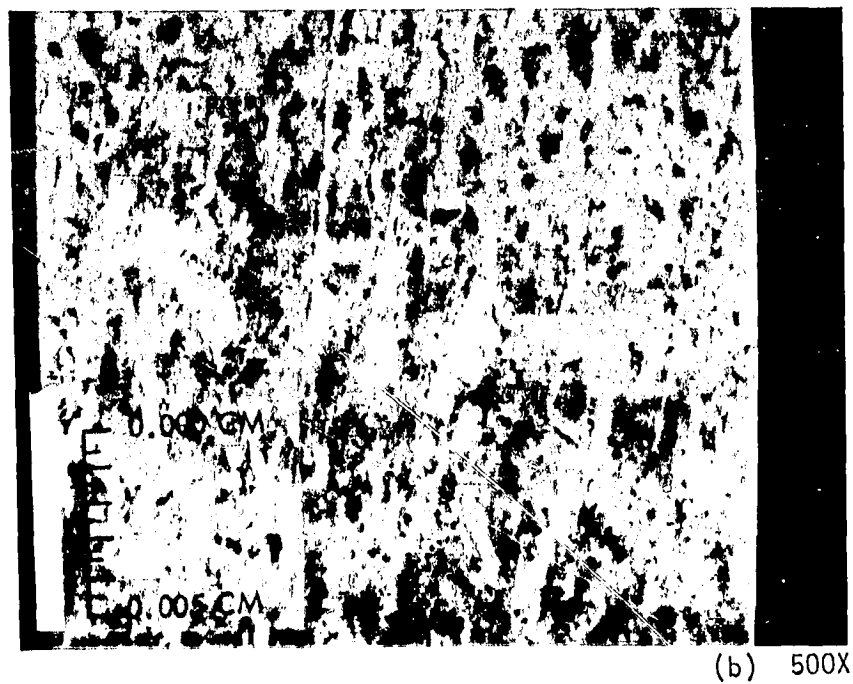
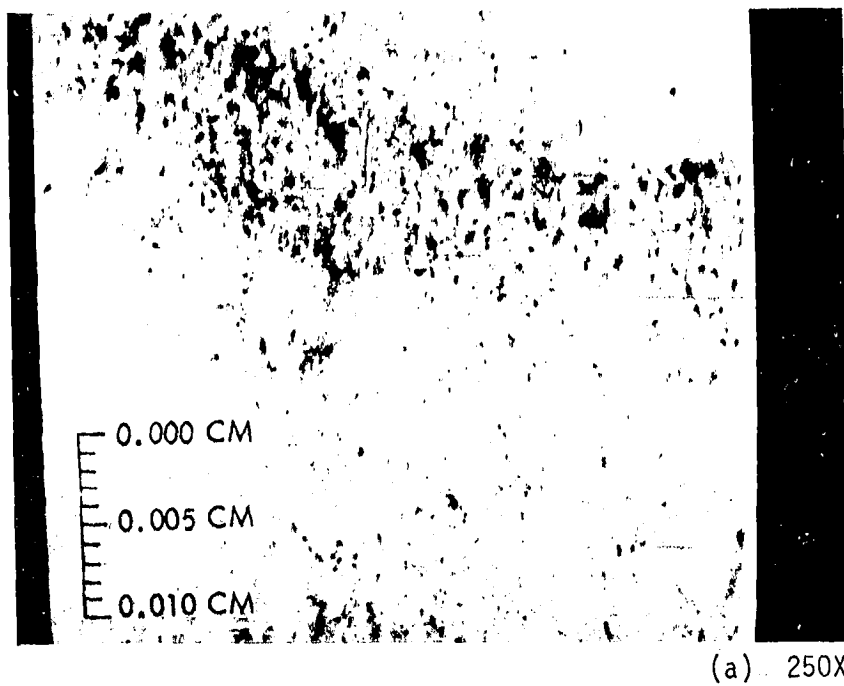


Figure 13. SEM Photo of Surface of Specimen 7234 Vapor Exposure

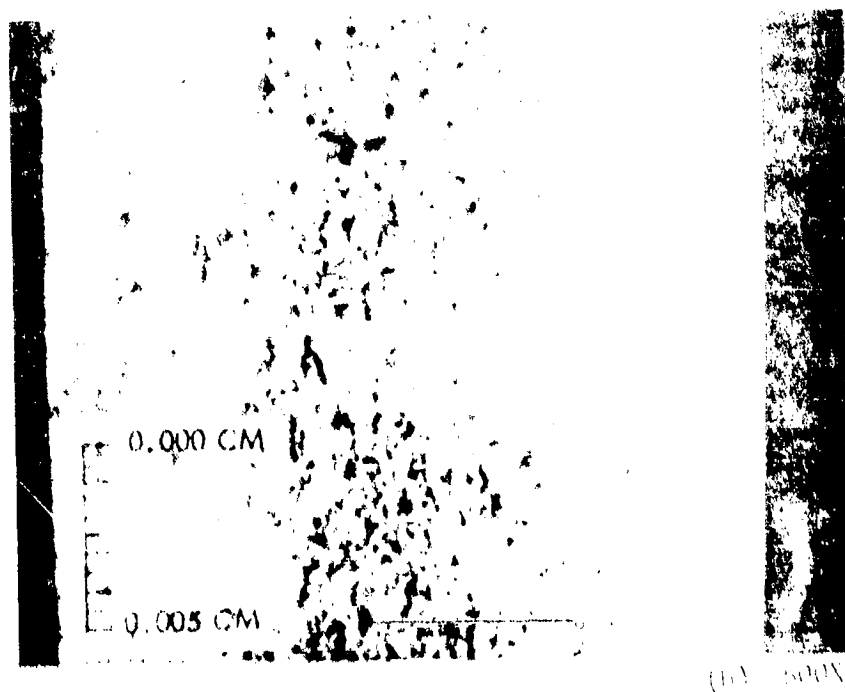
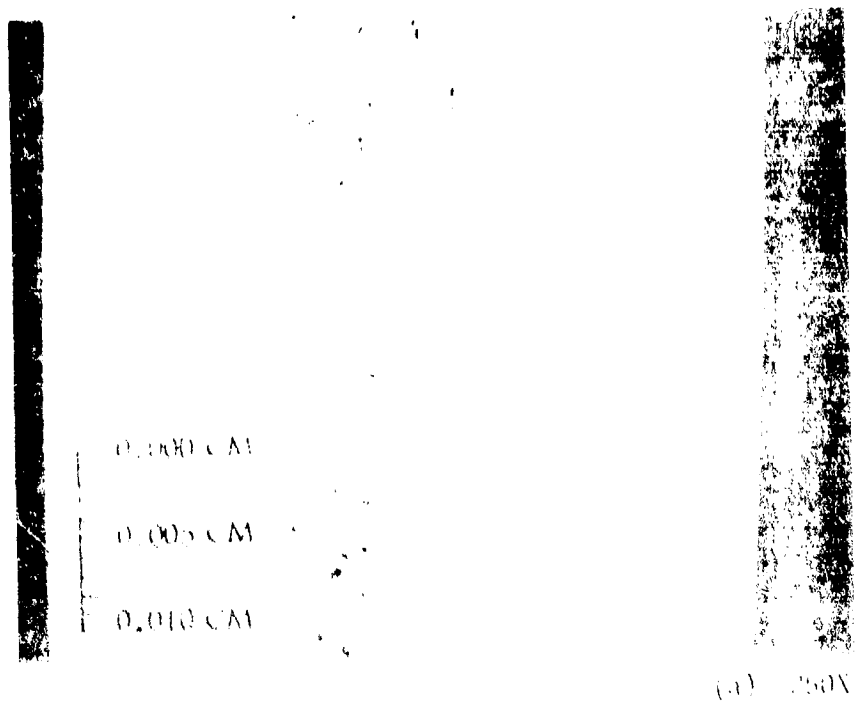
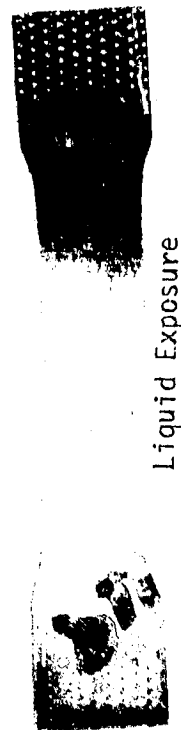
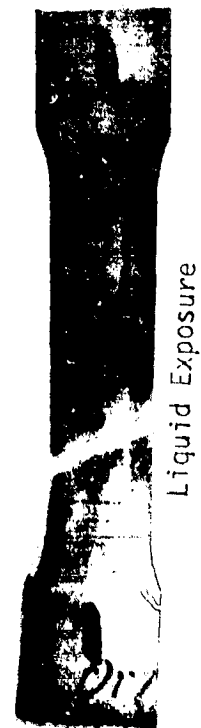
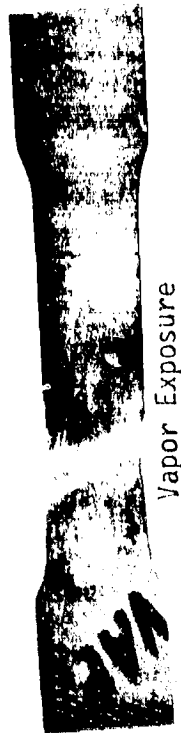


Figure 11. SEM Photo of Surface of Specimen 1000 Hours Exposure



(a) Specimen 7234 (1.3X) (b) Specimen 7214 (1.3X)

Figure 15. Tensile Tested Ti-6Al-4V Specimens

TABLE IV

TENSILE TEST RESULTS OF PROPELLANT EXPOSED SPECIMENS

Specimen	Propellant Exposure	Yield Strength, ksi 0.2% Offset	Ultimate Tensile Strength, ksi	Elongation % in 1-inch
7214 liq.	F ₂	140.0	149.2	13
7214 vap.	F ₂	139.8	150.0	13
7234 liq.	FLOX	141.6	150.0	13
7234 vap.	FLOX	138.7	149.6	14

The weights of the double dogbone specimens before the exposure to the propellants and after removal from the propellants are given in Table V.

TABLE V

WEIGHT OF SPECIMENS BEFORE AND AFTER

Sample	Propellant	Initial Weight, g	Final Weight, g	Weight Change, g
7214	F ₂	4.8850	4.8837	-0.0013
7234	FLOX	4.8892	4.8939	+0.0047

4.2.4 Metallographic Examination

Metallographic examination was performed on a cross section of specimen 7234 for the purpose of characterizing the corroded conditions for the liquid- and vapor-exposed surfaces. Samples representative of liquid- and vapor-exposed areas were cut and coated with electroless nickel to preserve the edges for polishing. The coated samples were mounted in epoxy and polished by conventional metallographic techniques. Metallographic analyses of pit frequency, depth and width were made and reported in Table VI for each successive plane at incremental polishing.

TABLE VI
PIT CHARACTERIZATION

Surface	Pit Concentration Number/cm ²	Pit Size/cm ²	Pit Depth/cm
Vapor-exposed	7×10^5	3×10^{-8} 2×10^{-7}	0.002 and below
Liquid-exposed	7×10^6	3×10^{-8} 2×10^{-7}	0.002 to 0.0005

It is obvious for this specimen that microscopic pitting of the liquid-exposed surface is more severe than the vapor-exposed surface. However, neither liquid nor vapor FLOX exposure indicated any significant corrosive attack on Ti-6Al-4V titanium for the specific test conditions and duration.

Typical surface profiles for liquid and vapor exposures are shown in Figures 16 and 17. It is evident from these photomicrographs that surface attack was so mild and general that it required high magnifications to resolve the pitting condition.

4.3 PHASE II - POST-TEST CHARACTERIZATION

Phase II consisted of opening four capsules after storage from 13 November to 26 August 1973 (9 months) containing propellant and double dog-bone specimens, removing the propellant for analysis, removing the specimens, analyzing propellant residues, if present, and submitting the specimens to mechanical testing and metallurgical analysis. The capsules removed were as follows:

- One (1) capsule containing aluminum and liquid fluorine
- One (1) capsule containing titanium and liquid fluorine
- One (1) capsule containing aluminum and liquid FLOX
- One (1) capsule containing titanium and liquid FLOX

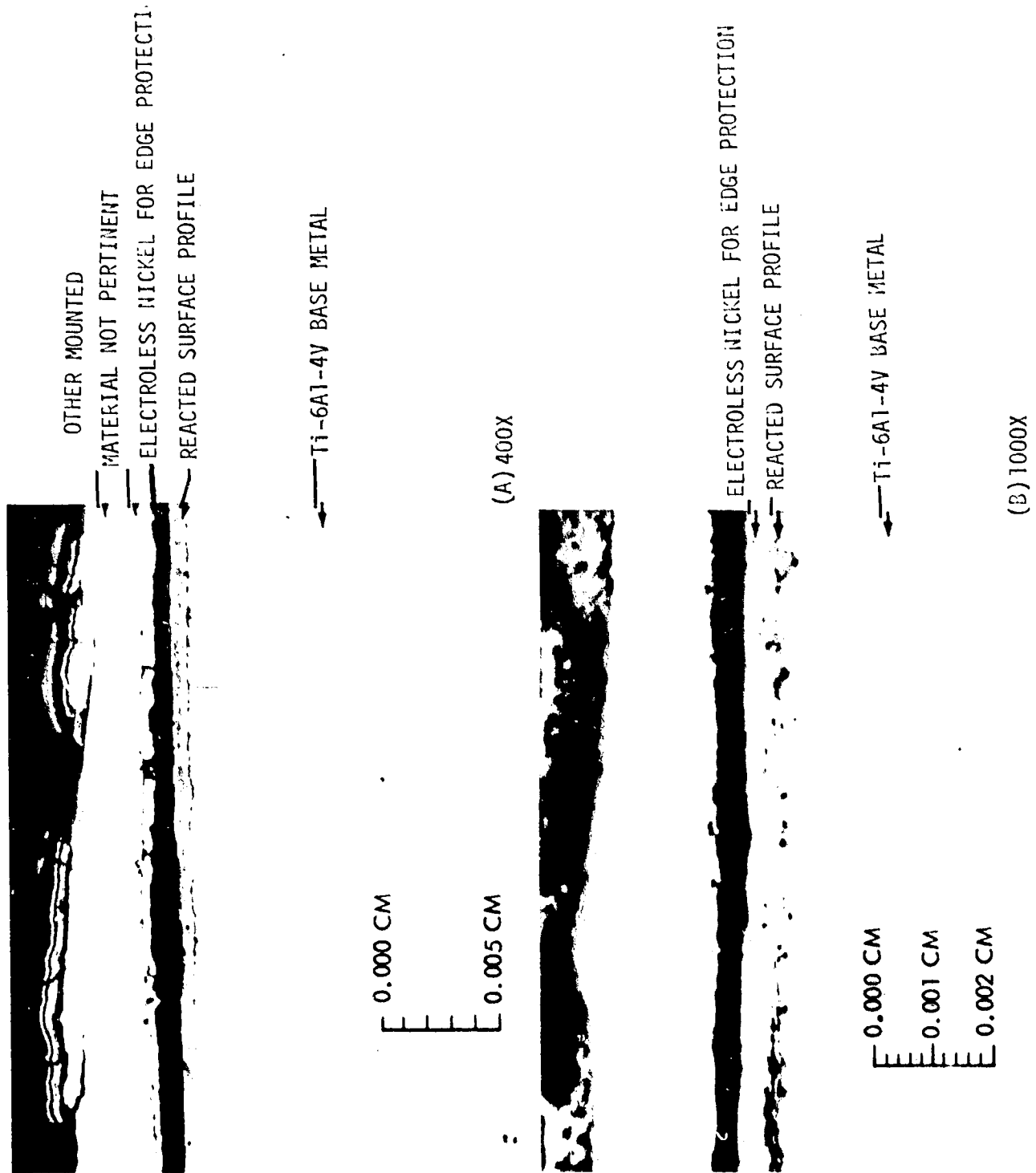
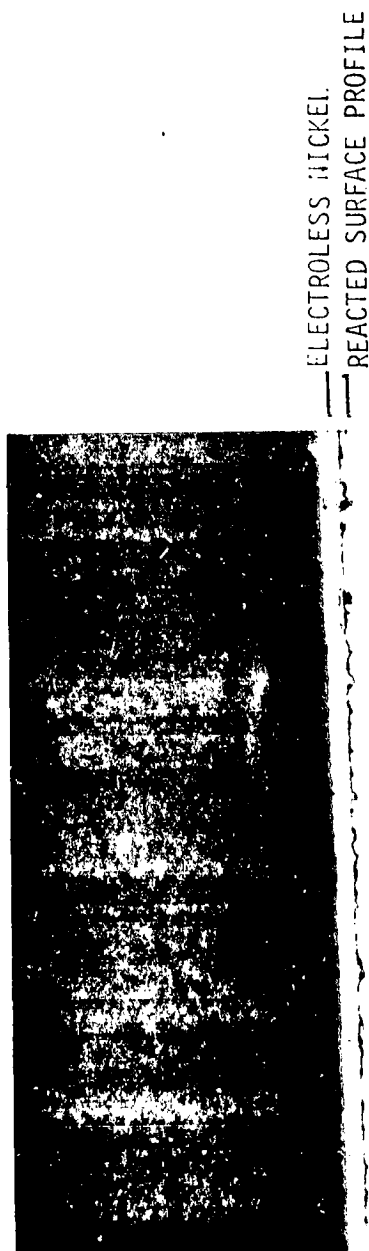
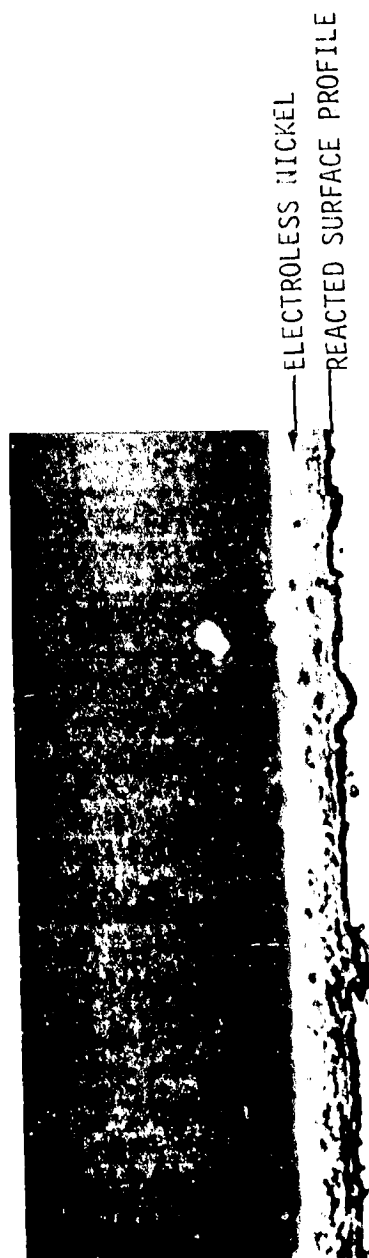


Figure 16. Cross Section of Vapor Exposed Part of Specimen 7234 Showing Reacted Surface Profile.



0.000 CM
0.005 CM

(A) 400X



0.000 CM
0.001 CM
0.002 CM

← Ti-6Al-4V BASE METAL

4.3.1 Characterization of Propellant and Residues

The capsules were opened according to the procedure described in the attached Program Plan. That is, the capsule was immersed about 80% in liquid nitrogen and then the tip of the capsule was removed in a vacuum environment whose total volume was 353 cc. The expansion of the vapors and the resulting removal of a small quantity of liquid phase into the vapor phase appeared to cause a slight drop in the propellant temperature. This conclusion was drawn from the fact that the pressure in the manifold connected to the capsule rose slowly over a considerable time period (about five minutes). The pressure observed upon opening all capsules, after a five-minute delay was 480 ± 10 torr.

The propellants were analyzed by gas chromatography and infrared spectrophotometry in the vapor phase upon opening the capsule and in the vapor phase after about one-half of the propellant had been distilled from the capsule while in a liquid nitrogen bath. When all the propellant had been distilled from the capsule, the capsule was allowed to warm to room temperature, the pressure in the vacuum system measured, and the total quantity of impurities determined by infrared spectrophotometric analyses. All analyses were performed according to the procedures described in the Program Plan attached to this report. The results of these analyses are tabulated in Table VII. The four compatibility capsules opened in Phase II post test characterization did not show any discernable etching of the surface thereby reconfirming the validity of the use of a glass capsule in these tests.

In a program conducted by the Boeing Company and funded by JPL (NAS7-789) analyses were made of the quantity of elements extracted from titanium and aluminum specimens submerged in liquid fluorine and FLOX for short periods. To provide a basis for correlation of data from this and the Boeing program, the quantity of fluoride and metallic elements (expected on the basis of specimen composition, Table VIII) extracted from the sample was determined. Careful visual examination of the evacuated capsules indicated the presence of a minor amount of scaling residue on either the specimen and none within the capsules. The titanium samples were slightly darkened but the aluminum samples appeared normal. After opening scratched capsules by contact with a hot glass rod, the gas and liquid exposed surfaces of the double dogbones, and the inside of the glass capsules were each washed

TABLE VII
COMPOSITION OF PROPELLANTS AND RESIDUE AT STORAGE TEMPERATURES

Sample No.	Metal	Propellant	Fluorine % (v/v)	Oxygen % (v/v)	SiF ₄ % (v/v)	Residue Composition (mg)
7208	Aluminum	Fluorine				
	Vapor on opening		100	0.0	0.0	
	Vapor for 0.5 distilled Residue		100	0.0	0.0	1.0 4.0 22.0
7210	Titanium	Fluorine				
	Vapor on opening		100	0.0	0.0	
	Vapor for 0.5 distilled Residue		100	0.0	0.0	3.4 0.8 4.0
7220	Aluminum	FLOX				
	Vapor on opening		85.7	14.0	0.3	
	Vapor for 0.5 distilled Residue		-	-	-	4.2 1.7 2.0
7244	Titanium	FLOX				
	Vapor on opening		93.2	6.8	0.0	
	Vapor for 0.5 distilled Residue		90.9	9.2	0.0	3.4 0.8 2.0

TABLE VIII
COMPOSITION SPECIMEN SURFACE WASHINGS

Sample No.	Sample Location	Initial Wt. g	Weight Loss, g	Extracted Weight, μ g						
				F	Al	Fe	Cu	Mn	Ti	V
7208	Al-F ₂ liquid Al-F ₂ gas Capsule	3.0726	-0.0011	150	200	187	1.5	4.5	-	-
				136	250	176	2.0	5.0	-	-
				24	85	4	2.5	0.5	-	-
7210	Ti-F ₂ liquid Ti-F ₂ gas Capsule	4.8647	-0.0012	65	40	47	-	-	<50 ^a	<3 ^a
				65	40	65	-	-	<50 ^a	<3 ^a
				28	40	11	-	-	<50 ^a	<3 ^a
7220	Al-FLOX liquid Al-FLOX gas Capsule	3.0910	-0.0007	105	175	88	1.7	1.5	-	-
				47	155	95	1.5	3.5	-	-
				43	<30	3	3.0	3.0	-	-
7244	Ti-FLOX liquid Ti-FLOX gas Capsule	4.8506	-0.0014	53	40	87	-	-	80 ^a	<3 ^a
				38	<30	65	-	-	<50 ^a	<3 ^a
				35	40	4.5	-	-	<50 ^a	<3 ^a

^aLimit of detection

with 10 ml of water adjusted to a pH of 7.5 using a 5-ml pipet to dispense the water. After washing a minor amount of residue was retained on the specimens. Because metallographic and SEM examination was to be performed, no other external means were employed to remove the adhering residue.

The fluoride ion was determined with an Orion fluoride ion electrode, Model 94-09, and the metallic ions determined by atomic absorption spectrometry using a nitrous oxide-acetylene flame for titanium, aluminum, and vanadium, and an air-acetylene flame for the other elements. The results of these analyses, initial specimen weights and weight losses are presented in Table VIII.

4.3.2 Visual and Microscopic Characterization and Tensile Testing

Visual and microscopic examination and tensile testing were performed in the same manner as the previous specimens. A sketch in Figure 18 shows the surface appearance of the four specimens analyzed. Except for some random staining in the Ti-6Al-4V specimens 7244 and 7210, none of the specimens showed any sign of surface attack.

Tensile test results of the liquid-exposed (L) and vapor-exposed (V) specimens taken from each of the double dogbone specimens are tabulated in Table IX. The yield strength, ultimate tensile strength and percent elongation of all titanium specimens are about equal and typical for unexposed Ti-6Al-4V sheet materials. Similar conclusions can be drawn for the 2219-T87 Al specimens. Therefore, it is concluded that the tensile properties of these materials are not affected by exposure to fluorine or FLOX either in the vapor phase or in the liquid phase.

4.3.3 Metallographic Characterization

Figure 19 (a) and (b) are photographs of the four double dogbone specimens after exposure. There was no sign of chemical attack on these specimens when examined at magnifications up to 40X. Figures 20 and 21 show tensile specimens all exhibiting a typical ductile 45° fracture. Evidently there was no embrittlement that resulted from exposure to either F_2 or FLOX. The same conclusion can be drawn from the tensile elongation data reported previously.

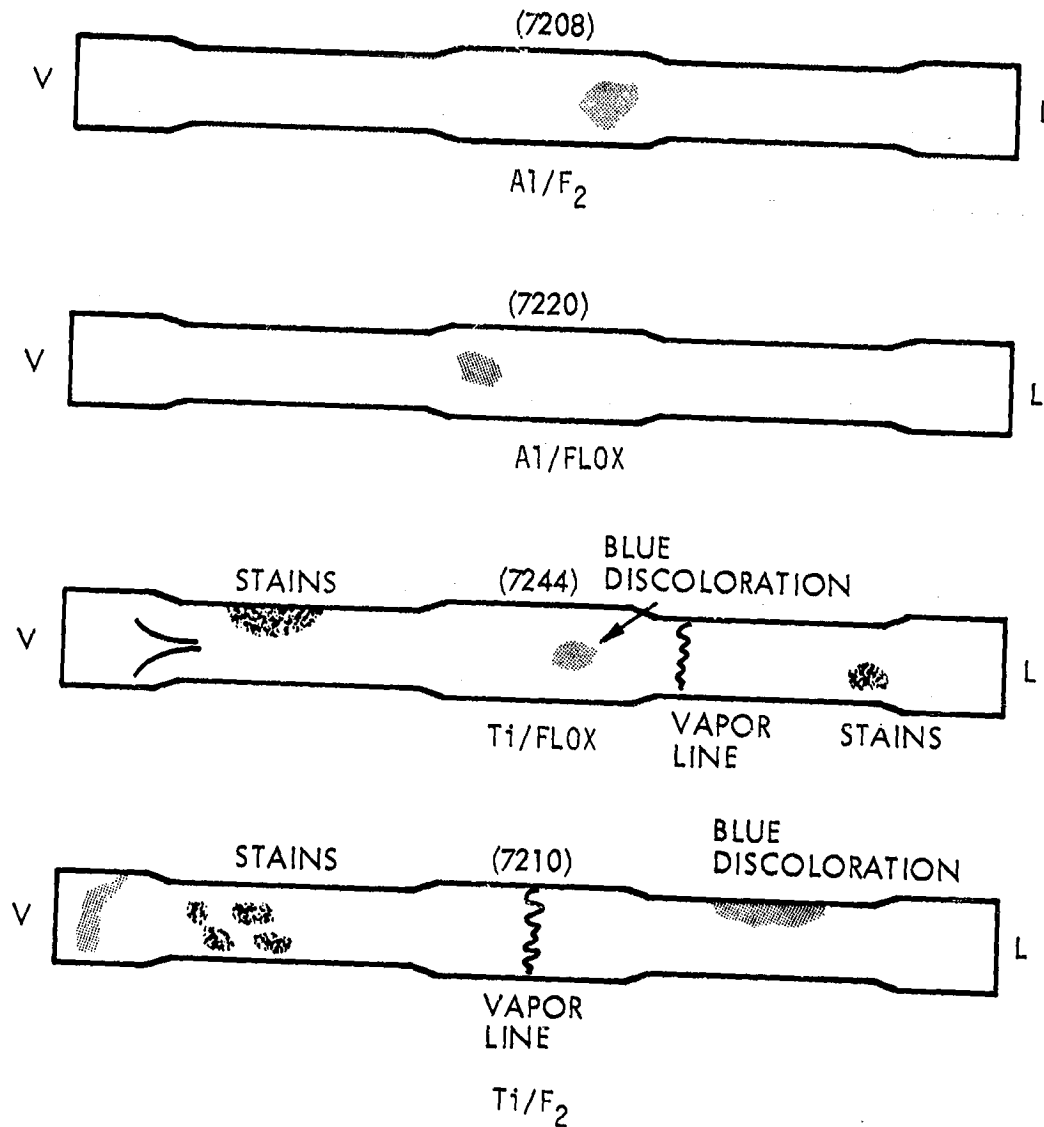


Figure 18. Mapping of Surface Appearance of Double Dogbone Specimens 7208, 7220, 7244 and 7210.
(V = Vapor End, L = Liquid End)

TABLE IX
TENSILE TEST RESULTS

Specimen	Propellant Exposure	Yield Strength, ksi 0.2% Offset	Ultimate Tensile Strength, ksi	Elongation % in 1-inch
7210L	F ₂	140.5	150.4	12
7210V	F ₂	139.8	148.8	12
7244L	FLOX	139.4	150.0	13
7244V	FLOX	140.3	149.6	12
7208L	F ₂	55.8	69.1	10
7208V	F ₂	55.8	69.4	10
7220L	FLOX	56.0	69.0	9
7220V	FLOX	54.9	68.4	10

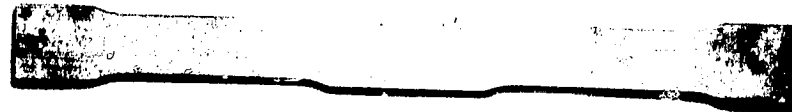
Results of quantitative analysis of pitting (Table X) were obtained by analyzing the scanning microscopic and metallographic data shown in Figures 22 through 29. It is evident from these results that pitting was more severe in the 2219-T87 aluminum alloy than in the Ti-6Al-4V titanium alloy for both F₂ and FLOX exposure. The degree of chemical attack is about the same in F₂ and FLOX environments for the aluminum alloy. However, a greater number of pits were observed in the FLOX-exposed specimen than in the F₂-exposed titanium specimen. In accordance with Champion's rating method for these specimens, the number of pits falls between "considerable number" and "very numerous," the size of pits is "minute" and the intensity (depth) of corrosion is "minute trace." One can conclude from these results, in conjunction with weight change data, that none of these materials exhibited deleterious effects in either F₂ or FLOX for the duration of the exposures.

#7220



Vapor

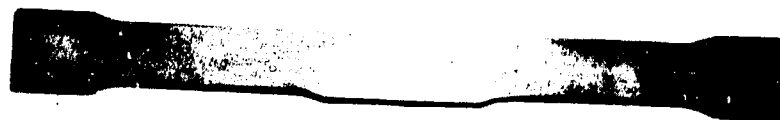
#7208



End



#7244



Vapor

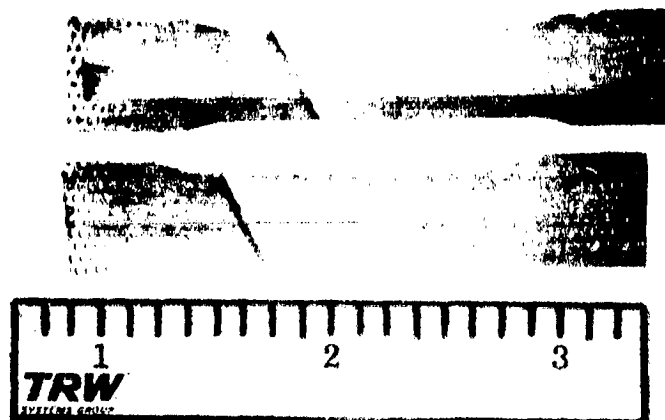
#7210



End

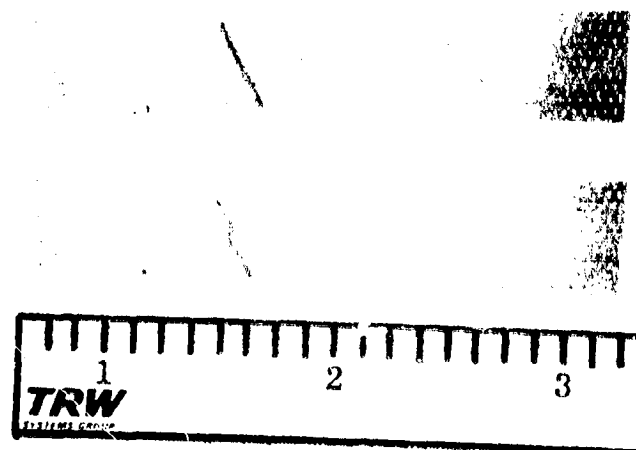


(b) Ti-6Al-4V Specimens
Figure 19. Double Dogbone Specimens After Exposure



#7220V

#7220L



#7208V

#7208L

Figure 20. Tensile Tested 2219-T87 Aluminum Specimens Showing Type of Fracture (1X)

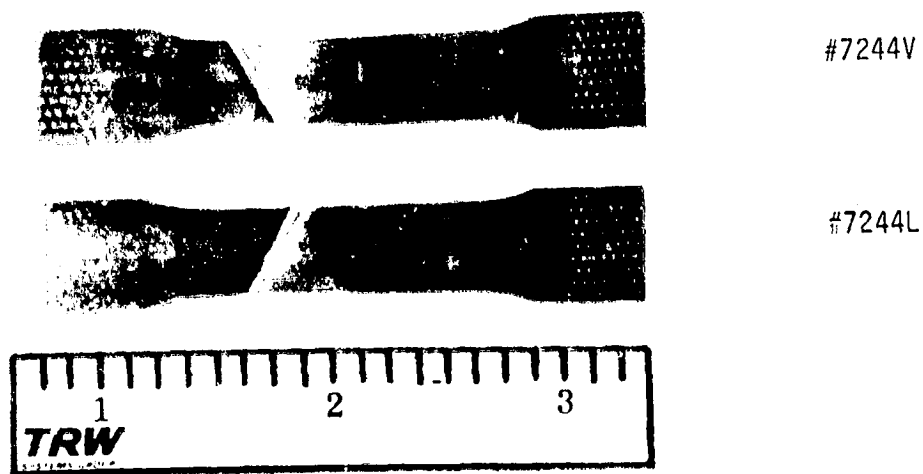
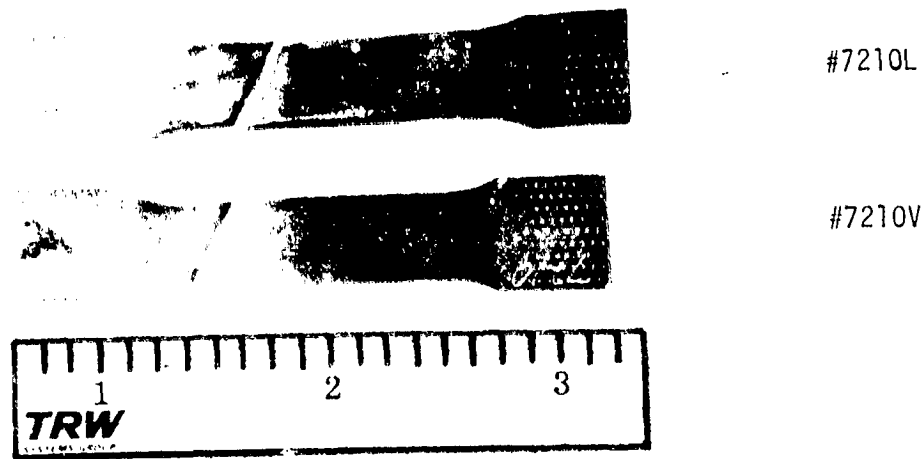


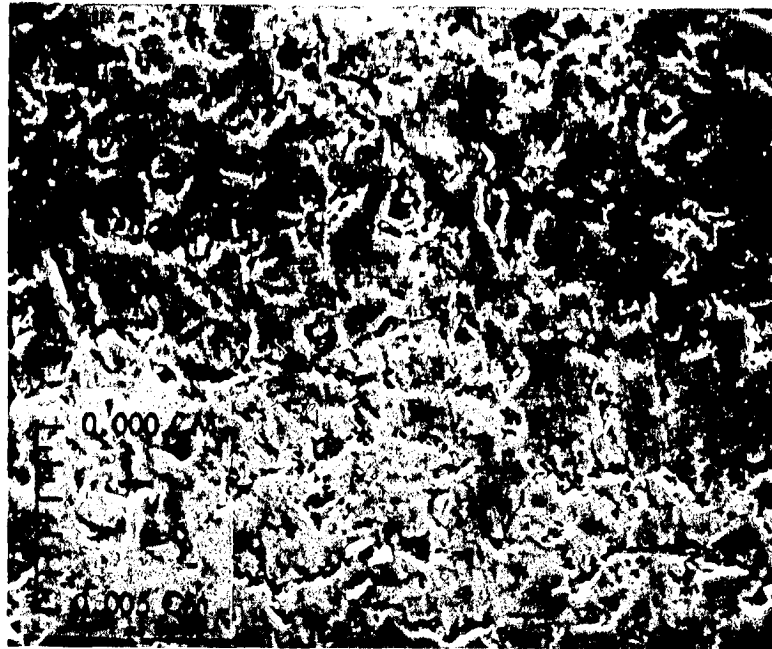
Figure 21. Tensile Tested Ti-6Al-4V Specimen,
Showing Type of Fracture (1X)

TABLE X
QUANTITATIVE ANALYSIS OF PITTING

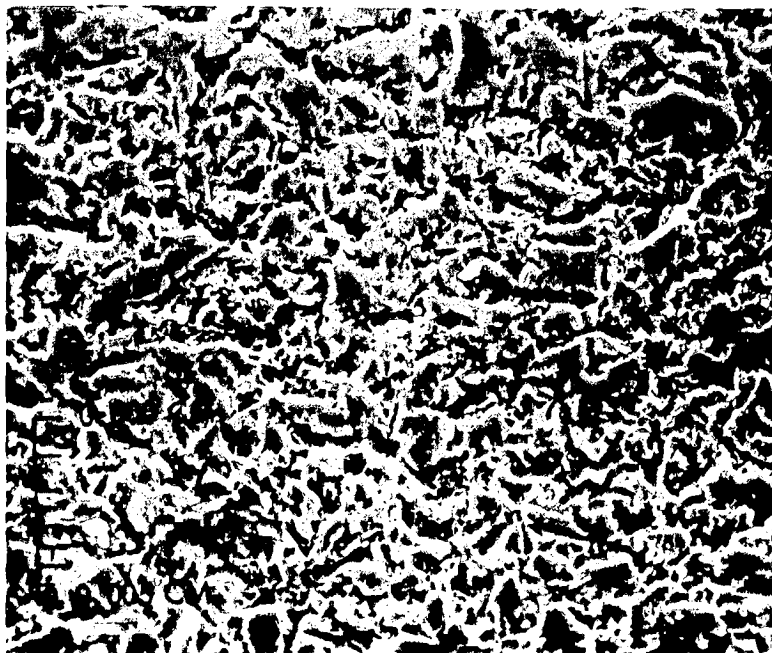
SPECIMEN	EXPOSURE MEDIUM	PIT CONCENTRATION, NUMBER/cm ²	PIT SIZE, cm ²	PIT DEPTH, cm
#7220 (Al)	FLOX (L)	6×10^5	4×10^{-7}	0.0004
	FLOX (V)	1×10^5	4×10^{-7}	0.0004
#7208 (Al)	F ₂ (L)	6×10^5	1×10^{-7}	0.0004
	F ₂ (V)	1×10^5	1×10^{-7}	0.0004
#7210 (Ti)	F ₂ (L)	1×10^4	1×10^{-8}	Below 0.0002
	F ₂ (V)	5×10^3	1×10^{-8}	Below 0.0002
#7244 (Ti)	FLOX (L)	3×10^4	1×10^{-8}	Below 0.0002
	FLOX (V)	2×10^4	1×10^{-8}	Below 0.0002

4.4 PHASE III AND PHASE IV - POST TEST CHARACTERIZATION

As indicated previously, the post test characterization of Phase III and Phase IV specimens for storage periods up to 70 weeks will be completed in May 1974. The results of these characterizations will be submitted as an addendum to this report.



(a) Vapor Exposure (500X)

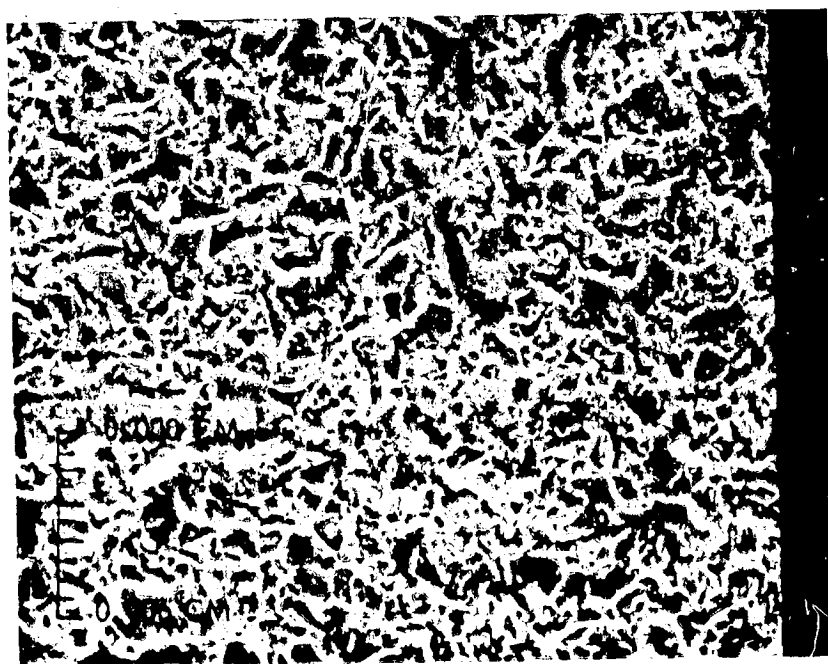


(b) Liquid Exposure (500X)

Figure 22. SEM Photos of 2218-T87 Aluminum Specimen #7220 After Exposure to FLOX

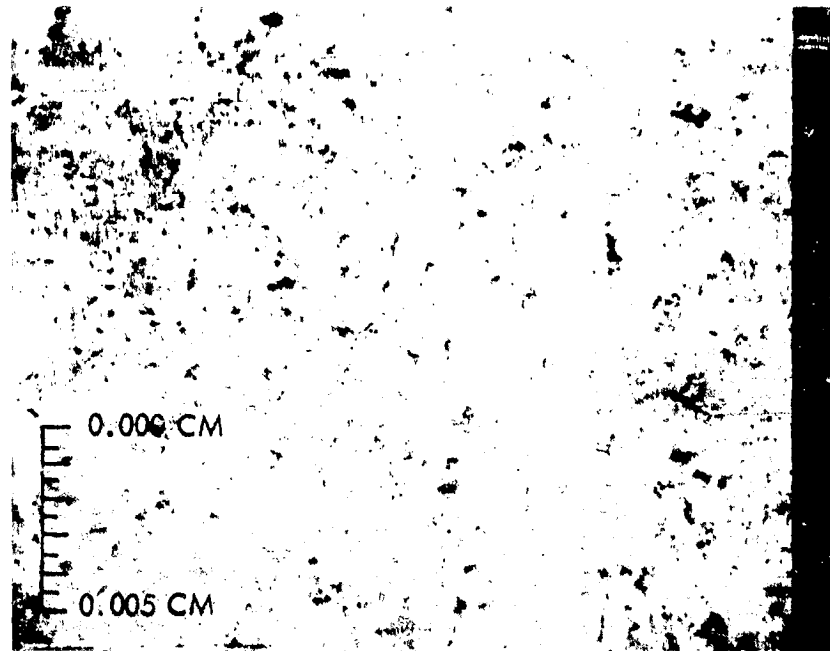


(a) Vapor Exposure (500X)

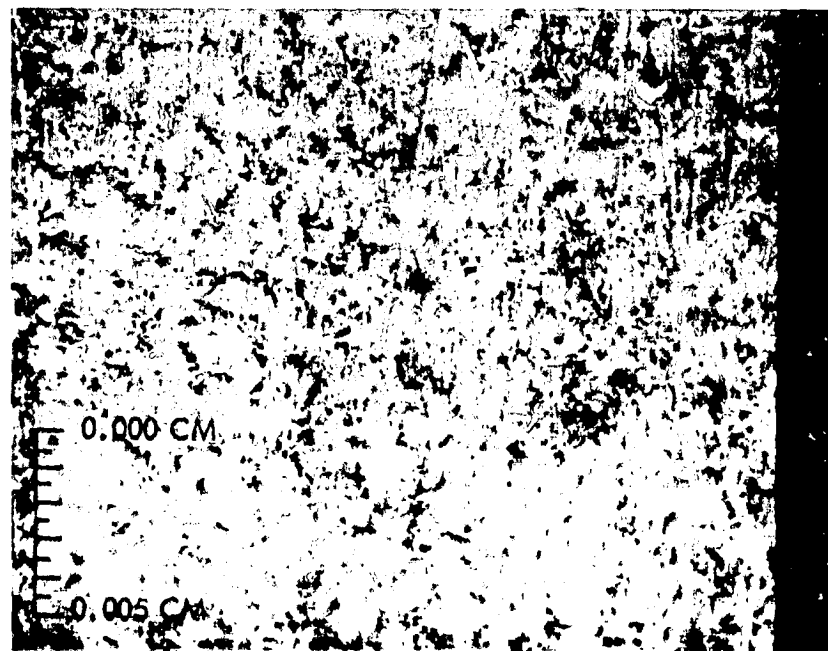


(b) Liquid Exposure (500X)

Figure 23. SEM Photos of 2219-T87 Aluminum Specimen #7208 After Exposure to Fluorine

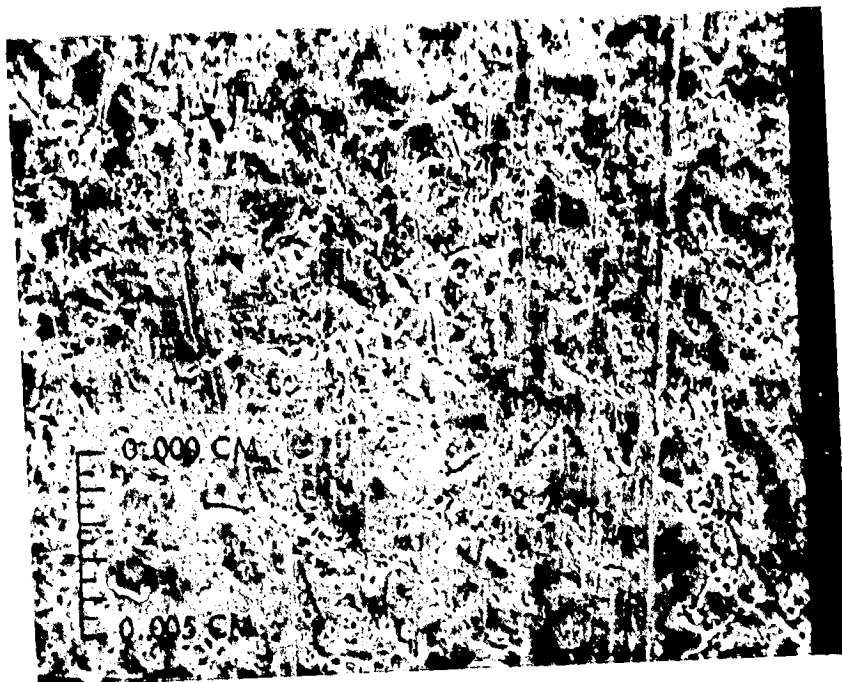


(a) Vapor Exposure (500X)

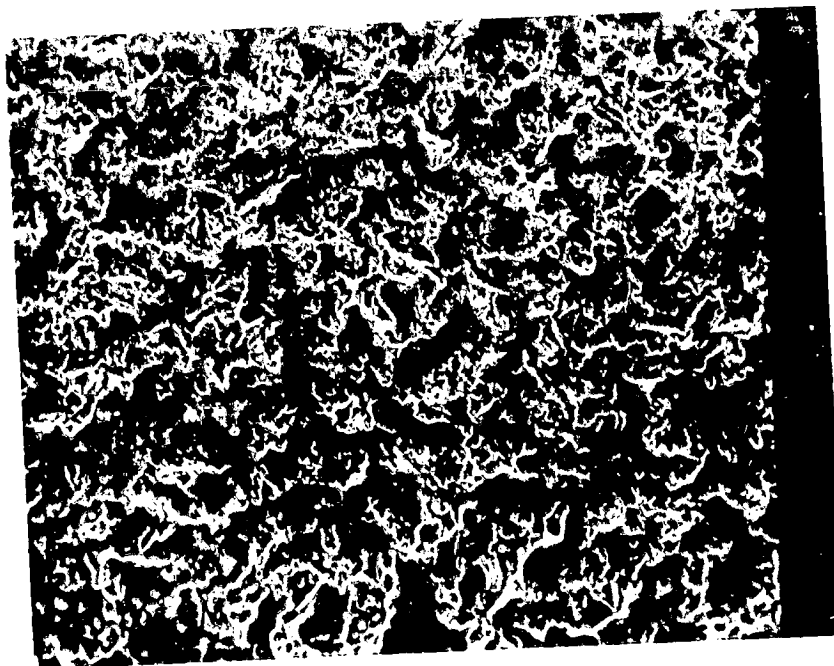


(b) Liquid Exposure (500X)

Figure 24. SEM Photos of Ti-6Al-4V Specimen #7210
After Exposure to Fluorine



(a) Vapor Exposure (500X)



(b) Liquid Exposure (500X)

Figure 25. SEM Photos of Ti-6Al-4V Specimen #7244
Surface After Exposure to FLOX



0.000 CM
0.005 CM



0.000 CM
0.005 CM

(b) Liquid Exposed (400X)

Figure 26. Cross-section of 2219-T87 Aluminum Alloy Specimen #7220 Showing Reacted Surface Profile

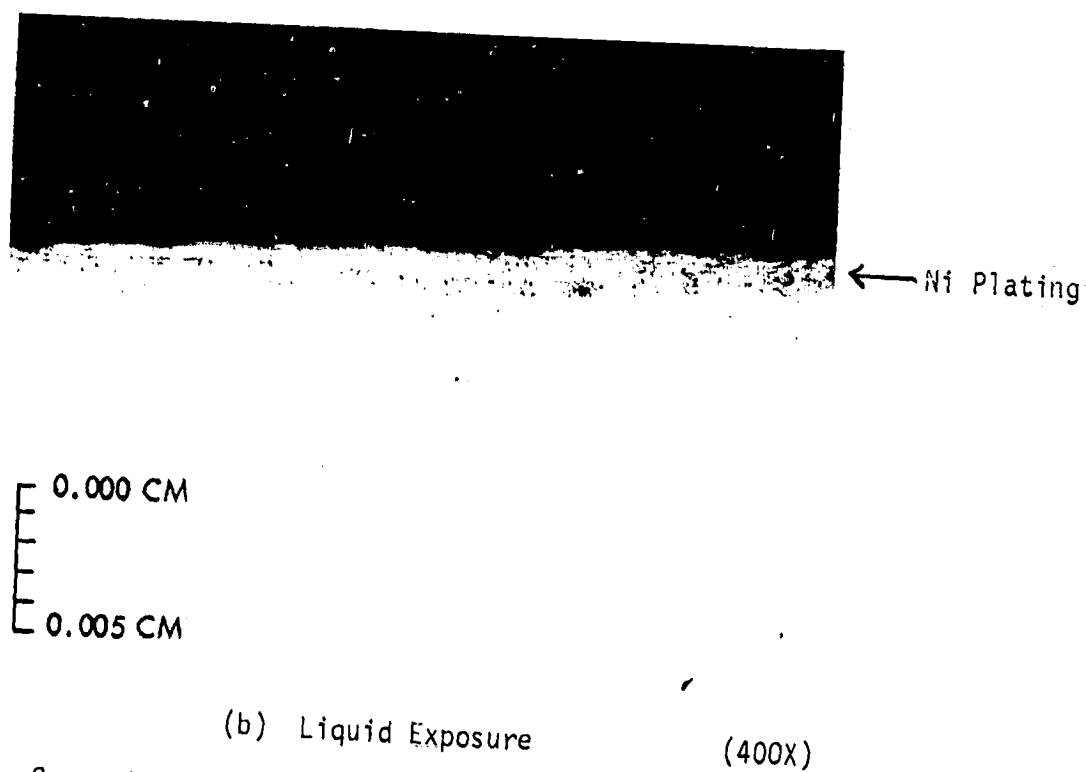
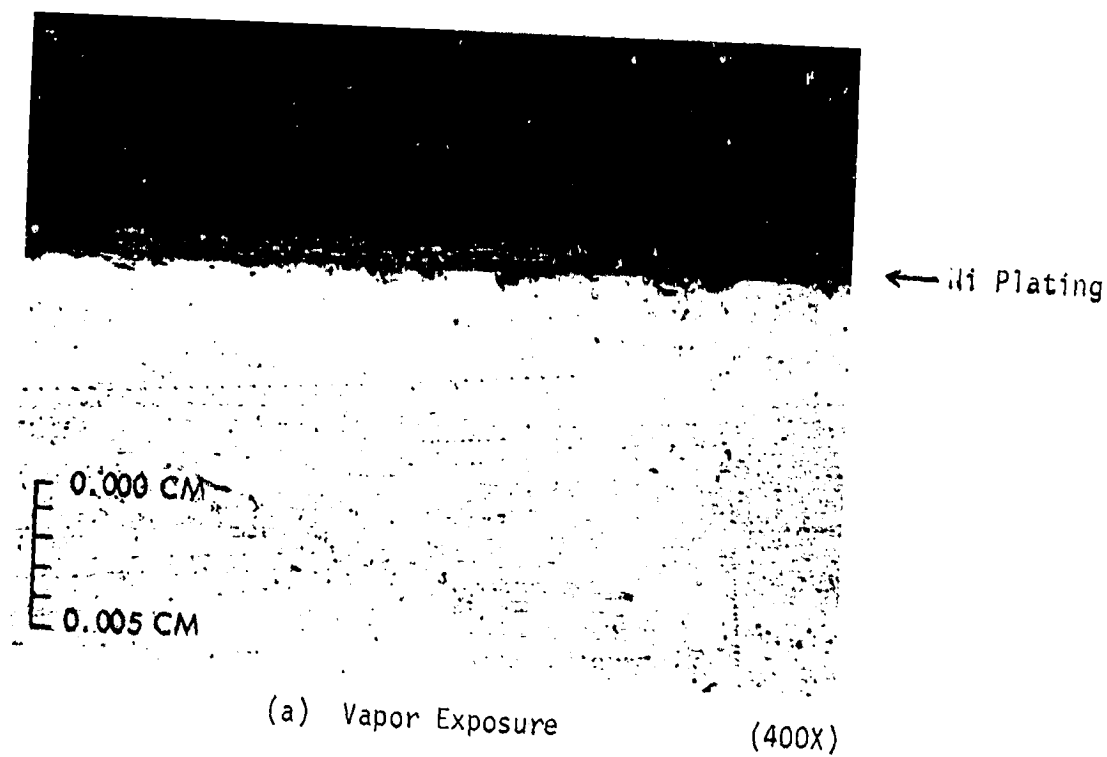


Figure 27. Cross-Section of 2219-T87 Aluminum Alloy Specimen #7208 Showing Reacted Surface Profile

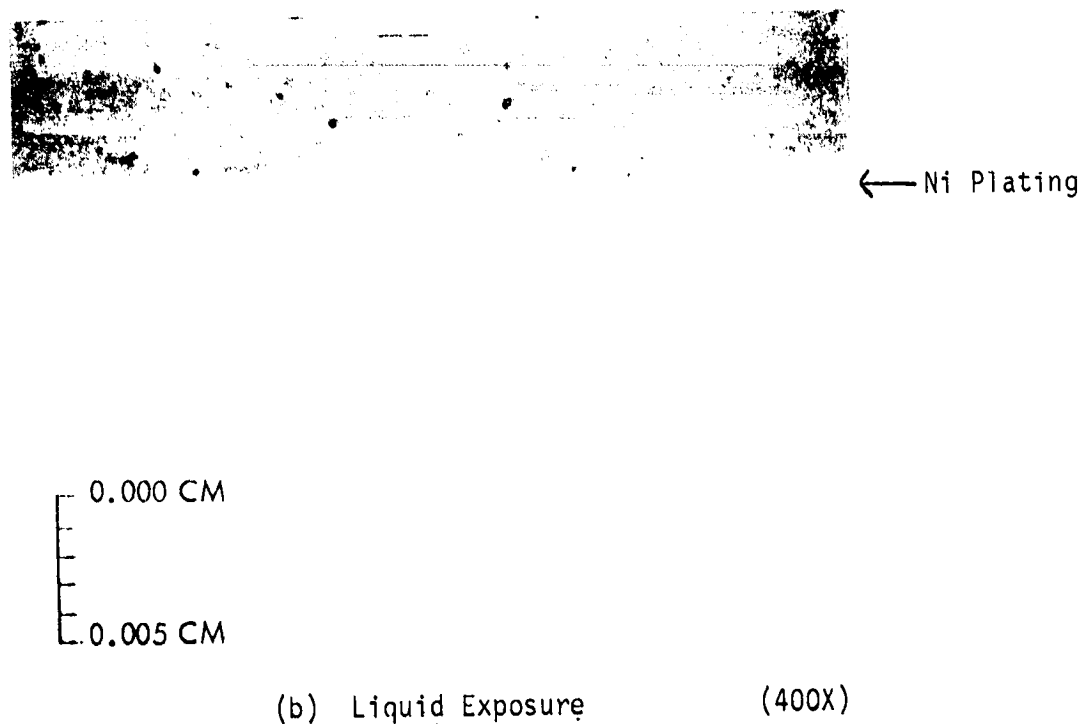
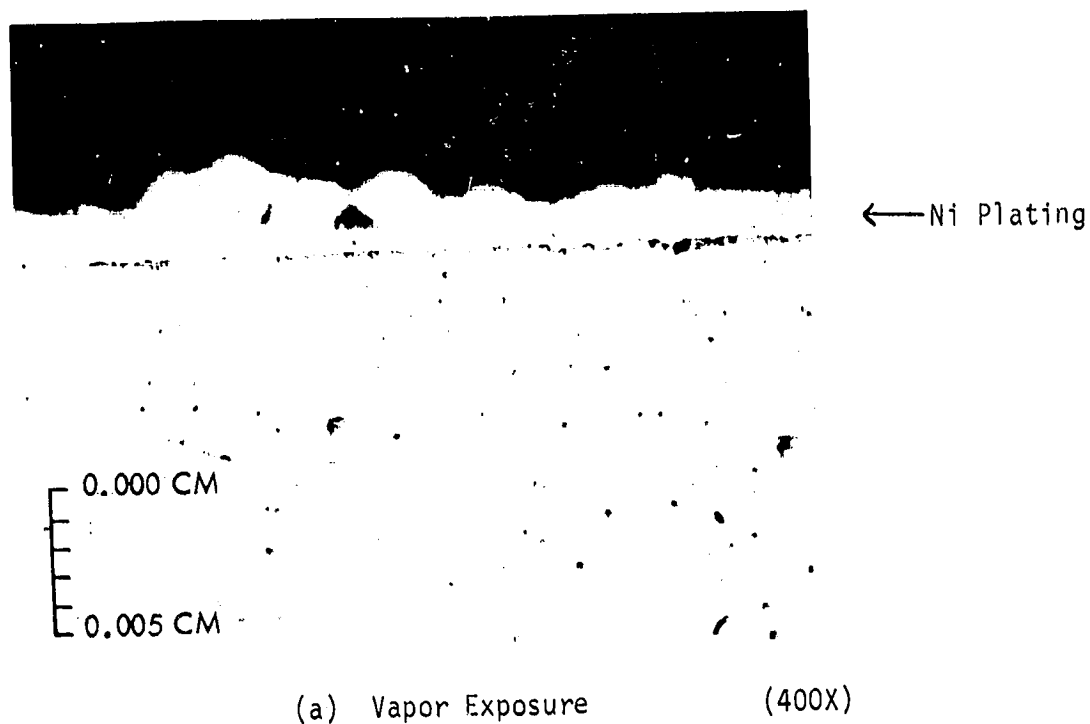
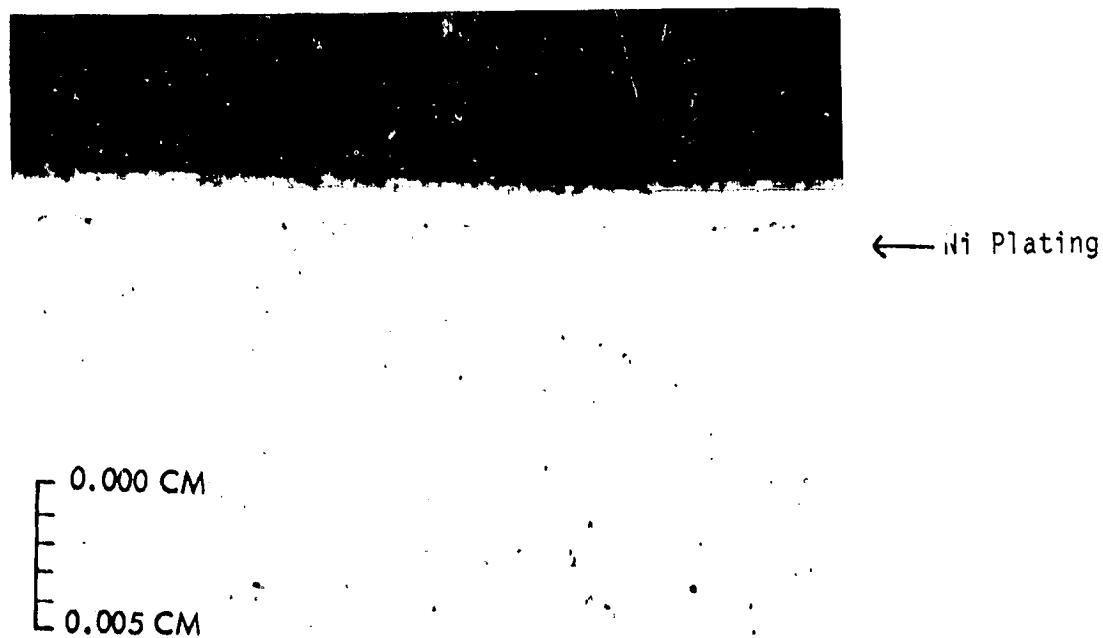


Figure 28. Cross-Section of Ti-6Al-4V Specimen #7244 Showing Reacted Surface Profile



(a) Vapor Exposure (400X)



(b) Liquid Exposure (400X)

Figure 29. Cross-Section of Ti-6Al-4V Specimen #7210
Showing Reacted Surface Profile

5. CONCLUSIONS AND RECOMMENDATIONS

As indicated in Section 4.3, preliminary findings after nine months storage have shown that a small degree of chemical attack occurred during storage of the aluminum and titanium alloy in the presence of FLOX and fluorine liquid propellants. The following general trends were observed:

- Pitting was more severe with the 2219-T87 aluminum alloy than with the titanium 6Al-4V alloy for both propellants.
- The degree of chemical attack is more severe in the presence of FLOX than in liquid fluorine.
- The degree of chemical attack is more severe in the liquid phase than in the vapor phase.
- The mechanical properties were not affected by storage in either of the propellants during this period of time.

It appears the use of the scanning electron microscope and counting of the pits number, pit size and pit depth is an excellent technique for discerning small microscopic corrosion effects. During the course of the post test characterization it became obvious that several procedural methods required standardization to ensure meaningful comparisons were obtained, e.g., 1) the use of a magnification of 400X to 500X is mandatory to discern some of the subtle differences and 2) standardization of the technique for counting and measuring the pit size and number.

The findings of this program, particularly the differences between the 36-week and 41-week storage durations provide the basis for the recommendation to extend the storage duration to provide clear-cut definition of the affects of storage time. It is important to see whether the observed corrosive phenomena rate levels off or accelerates as a function of time. Only in this fashion will unequivocal evidence be obtained to make valid extrapolations for hardware commitments of advanced missions to outer planets.

APPENDIX A
COMPATIBILITY TESTING DATA REQUIREMENTS

A.1 FLUID CHARACTERIZATION

A.1.1 Pretest

A.1.1.1 Identity and Source - Provide the following: manufacture and/or supplier, specification number used in the procurement, chemical name, chemical formula, trivial and/or code names, date of purchase and/or preparation, type of transporter and container material.

A.1.1.2 Chemical Analysis - Provide the following: major and minor components as provided by the manufacturer and/or determined by the user (report in weight percent), analytical method used by manufacturer and/or user, number of analysis replicates.

A.1.1.3 Impurity Analysis - Provide the following: soluble and insoluble components including particle size (report in weight percent). For cryogenic fluids, differentiate between particles which are solid at ambient conditions and those which melt or vaporize at ambient.

A.1.1.4 Storage History and Reanalysis - If a significant time has elapsed between fluid analysis as conducted under A.1.1.2 and A.1.1.3 and fluid use in testing, provide the following: storage history of the fluid and reanalysis as detailed under A.1.1.2 and A.1.1.3.

A.1.2 Post Test

A.1.2.1 Chemical Analysis - Provide the following: major and minor components (report in weight percent), analytical method used, indicate changes if any, from A.1.1.2 (report in weight percent), number of analysis replicates.

A.1.2.2 Impurity Analysis - Provide the following: soluble and insoluble components including particle size (report in weight percent), indicate changes, if any, from A.1.1.3 (report in weight percent).

A.1.2.3 Material-Fluid Interaction Products - Provide the following: identify the soluble and insoluble gas, liquid and/or solid constituents which may have been formed during the test (report in weight percent). If possible, report the rates of formation of these constituents.

A.1.3 Control Test for Fluid

A.1.3.1 Chemical Analysis - Provide the following: data as in A.1.2 for fluid exposed to same chemical, thermal and pressure environment or cycles as that for test but in the absence of material, number of analysis replicates.

A.1.3.2 Impurity Analysis - Provide the following: data as in A.1.2.2 for fluid as tested in A.1.3.1.

A.1.3.3 Decomposition Products - Provide the following: data as in A.1.2.3 for fluid as tested in A.1.3.1.

A.2 MATERIAL CHARACTERIZATION

A.2.1 Pretest

A.2.1.1 Identity and Source - Provide the following for either a metal or non-metallic material: manufacturer (including heat or batch, processing method (forging, plate extrusion, casting, molding, etc.) and mill (surface) finish, manufacturer's specification, MIL or AMS specification, trade name, chemical name, and chemical analysis (major and minor components).

A.2.1.2 Specific Processing - Provide the following: cleaning prior to preparation of specimen (including materials used), welded or unwelded (if welded, specify whether chill bars were used (include type of bar), type of welding (EB, TIG, MIG, etc.), and weld filler material used), heat treatment (i.e., stress relief prior to welding, after welding and, if not welded prior to test, any thermal processing), specimen configuration and orientation (transverse or longitudinal), cleaning after preparation (specify materials used and whether passivated, abrasive, etc.), surface finish and lay, and applied stresses or unstressed (if stressed, specify procedure, loads and specimen configuration).

A.2.1.3 Properties - Provide the following: surface area exposed in test, mechanical properties (i.e., tensile, ultimate, % elongation, etc.), metallographic properties, visual and magnified surface examination (specify magnification), map or record defect locations, and specimen weight.

A.2.2 Post Test

A.2.2.1 Visual Examination of Specimen - Provide the following: appearance, magnification, and time elapsed after test to examination with description of exposure environment.

A.2.2.2 Chemical Analysis of Surface Films - Describe corrosion products, etc., and specify type of analysis used.

A.2.2.3 Sample Weight - Provide the following: weight before surface films, corrosion products, etc., were removed and weight after removal of surface films, etc., (specify how surface films, etc., were removed).

A.2.2.4 Metallographic Examination - Provide the following: surface finish, cross section, and photographs of typical sections including magnification (including a standard scale in all original photographs), and photograph of views from a scanning electron microscope.

A.2.2.5 Mechanical Properties - Provide tensile, ultimate, % elongation, etc.

A.2.3 Control Test for Material

A.2.3.1 Inert Atmosphere Storage Without Stress - Specimen qualified under A.2.1 but stored in an inert atmosphere during compatibility test. Processed as in A.2.2 after test completion.

A.2.3.2 Inert Atmosphere Storage with Stress - Specimen qualified under A.2.1 and exposed to the same thermal and pressure environment or cycle in an inert atmosphere but in absence of fluid. Processed as in A.2.2 after test completion.

A.3 TEST PROCEDURES

A.3.1 Test Description

Provide a complete, detailed description of test method used. Refer to number and name of related standard test procedure.

A.3.2 Test Precautions

Provide a complete, detailed description of the precautions exercised to eliminate spurious test results.

A.3.3 Temperature and Thermal Cycle

Provide data in English and System Internationale (Metric) system units.

A.3.4 Pressure

Provide data in English and S.I. (Metric) system units.

A.3.5 Exposure Duration

A.3.6 Stress Level

Provide data on stress level (at test temperature) and method used for its measurement. Report any changes during the test period.

A.3.7 Geometry

Provide surface to volume ratio and other geometric effects.

A.3.8 Flow Rate

Provide data in English and S.I. (Metric) system units.

A.3.9 Instrumentation

Provide information on the instrumentation used and data as to its accuracy and precision.

A.3.10 Ullage Volume

Provide data in absolute and relative volumes.

APPENDIX B

PROCEDURE FOR PREPARATION OF FLOX

B.1 SCOPE

This procedure describes the preparation of FLOX (88/12 F₂/O₂ mixture by weight).

B.2 PURPOSE

The purpose of this procedure is to provide sufficient information in order to prepare a FLOX blend in a safe manner.

B.3 FLOX PREPARATION

Connect with clean passivated stainless steel or brass tubing and fittings a fluorine tank (0.5-lb F₂) to a gauge and to a line running to an oxygen tank. After the pressure in the fluorine tank has been measured, the valve on the fluorine tank is closed. Then open the oxygen supply tank and adjust by means of a regulator the pressure to the value indicated by the Equations 1 and 2. Then open the valve on the fluorine tank and allow the pressure in the fluorine tank to equilibrate. Immediately close the valve on the fluorine tank. Then by means of the procedure described in Appendix F analyze the fluorine-oxygen (FLOX) mixture to determine that the composition is the desired 88/12 fluorine-oxygen mixture.

$$T_p = \frac{PF_2}{0.8606} \quad (1)$$

$$PO_2 = TP - PF_2 \quad (2)$$

Where: T_p = total pressure, psia

PF_2 = pressure in fluorine tank, psia

PO_2 = pressure of oxygen to be added, psia

0.8606 = mole fraction of fluorine in a 88/12 F₂O₂ mixture by weight

Note: This operation is dangerous. All valves must be opened and closed either by means of remotely operated valves or by extensions through a

23162-6020-RU-00

barricade. Adequate provisions must be made to handle a fire that consumes all the fluorine present. Lines must contain check valves to prevent the flow of fluorine into the oxygen tank. Before disconnecting, all lines must be purged with nitrogen.

APPENDIX C

PROCEDURE FOR PREPARATION OF TEST SPECIMENS

C.1 SCOPE

This procedure describes the preparation of test specimens; both metallic and non-metallic.

C.2 PURPOSE

The purpose of this procedure is to provide sufficient information in order that test specimens can be manufactured by shop personnel.

C.3 METALLIC SPECIMENS

C.3.1 Double Dog-Bone SpecimensC.3.1.1 Weld or Brazed Specimens

Obtain sheet stock approximately 0.040-inch thick. Weld or braze 1.3-inch wide strips of the two sheet stocks, making the joint traverse to the rolling direction. Cut (as described in Paragraph B.3.1.2) the jointed materials in such a manner that the joint traverses the gauge section. X-ray welds or brazed section of specimens and retain X-ray with Test Data Sheet.

C.3.1.2 Standard Specimen

Cut a slightly oversized specimen (Figure C.1) with a band saw, using sheet material that is approximately 0.05-inch thick. Mill width and length dimensions to those shown in Figure C.1. Rough grind and then lap thickness to the requirements of Figure C.1. To eliminate contamination during grinding, dress grinding wheels, completely clean machines and use new coolant. Use masks thinner than the specimen to hold the specimen in place. Dimensionally inspect specimens for compliance with the drawing (Figure C.1). Vibrate etch serial number on each specimen. Initiate a Test Data Sheet for each specimen. Clean the specimen in accordance with JPL Process Specification FS506300, Revision A, dated 26 October 1971, "General Cleaning Requirements

for Liquid Fluorine and FLOX Propulsion Subsystem and Support Equipment Detail Specification For." The instructions in Section 3.10.5 Newly Machined Parts shall be followed. Before sealing the specimen in a plastic bag weigh the specimen and perform a visual and magnified surface examination, and map or record defect locations.

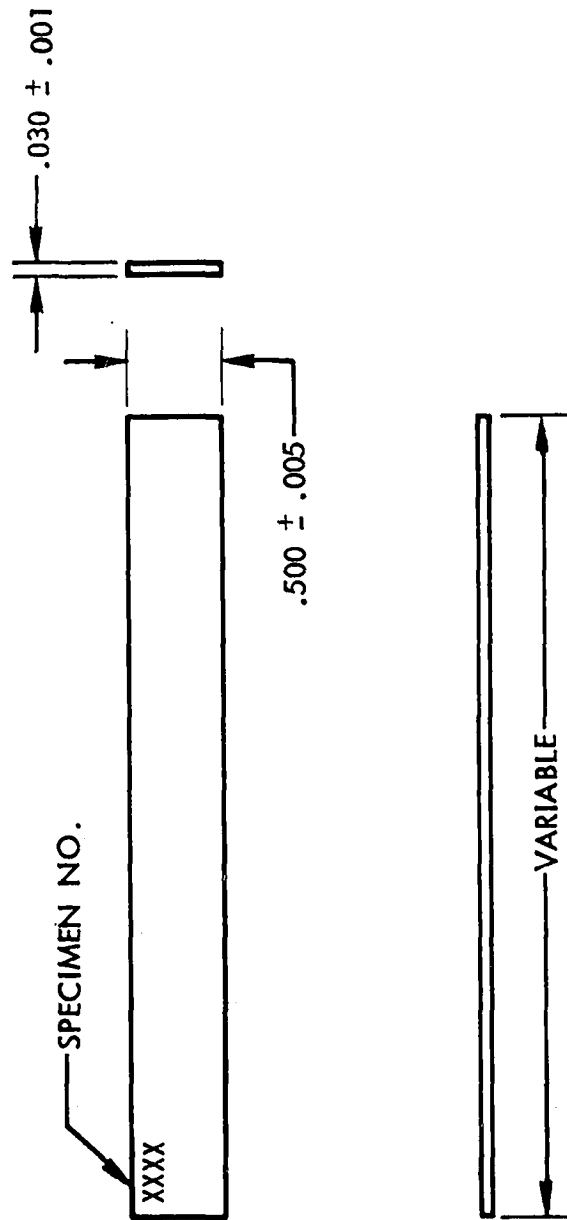
C.3.2 Non-Metallic Specimens

Prepare a die for cutting non-metallic specimens to the dimensions shown in Figure C.1. Cut molded sheets of the non-metallic specimens. The thickness of the sheets should be about 0.12-inch. Degrease the specimens with acetone. Then in a clean-room wash the specimens in a 0.4% of liquid detergent ("Joy"), at $180 \pm 30^\circ\text{F}$ for five (5) minutes. Then rinse thoroughly with distilled water. The pH of the run-off water must be equal to that of the initial distilled water.

C.3.3 Stressed Specimens

Calculate the required length of the stressed specimens using data collected from measured physical properties determined for Standard Test Specimens and the procedure described in Section 4.1 of the Final Report. Then cut with a band saw, a blank size of 0.062-inch x 0.625-inch and the calculated length + 0.19-inch. Mill length and width to the dimensions shown in Figure C.2. Dimensionally inspect specimens for compliance with the drawing. Initiate a Test Data Sheet for each specimen. Designate the material and lot or heat numbers on the Test Data Sheet. Clean the specimens in accordance with the instructions in JPL Specification FS506300, Revision A, Section 3.10.5 Newly Machined Parts. Weigh specimen and record weight on Test Data Sheet. Prepare a stress apparatus from the same material as the specimen to be stressed. The stress apparatus is to be manufactured according to Figure C.3. Degrease the stress apparatus with acetone. Then place the specimen to be stressed in the apparatus as shown in Figure C.3, being careful not to over stress the specimen while placing it in the stress apparatus.

In a clean-room, wash the specimen and stress apparatus before stressing the specimen in 0.4% non-ionic detergent, at $180 \pm 30^\circ\text{F}$ for five (5) minutes. Then rinse the assemblies thoroughly with distilled water until the pH of



NOTE: 1. BREAK ALL EDGES .005
 2. SURFACE .0025 FLAT/IN. TYP.
 3. ALL SURFACE 16 - 8

Figure C.2. Stressed Test Specimen

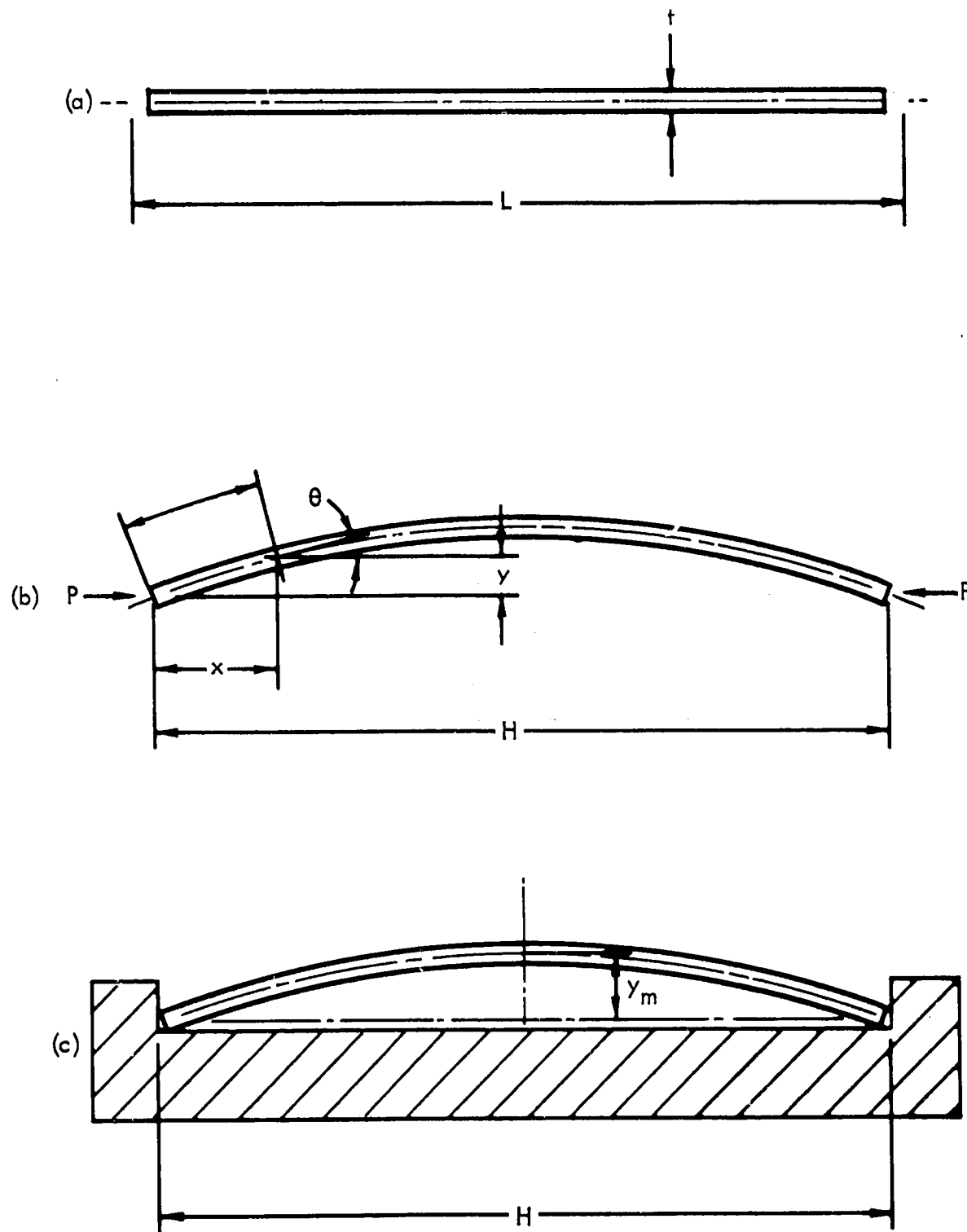


Figure C.3. Stressing of Bent-Beam Stress Corrosion Specimen

23162-6020-RU-00

run-off water is equal to that of the initial distilled water. Dry the specimen with dry filtered nitrogen gas. Place the specimen and stress apparatus in a clear clean plastic bag marked with the serial number of the specimen. Care must be taken to avoid hand contact after the cleaning operation. White gloves should be used to handle specimens.

APPENDIX D

PROCEDURE FOR MANUFACTURING OF TEST CAPSULES

D.1 SCOPE

The scope of this procedure includes methods used for test capsule manufacture, annealing, special cleaning, inserting specimens, glass fusing and packaging.

D.2 PURPOSE

The purpose of this procedure is to document the methods used to manufacture test capsules and to seal test specimens in the capsules.

D.3 CAPSULE MANUFACTURE

D.3.1 Capsule Section

Use a hydrogen-oxygen flame to perform all glass-blowing operations. This is to avoid leaving any carbon particles on or in the glass.

Prepare the capsule section (bottom section) to the dimensions shown in Figure D.1.

Prepare two glass rods, 0.3-inch in diameter by 2.5-inch long; for each capsule section (bottom section), except for those to contain stressed specimens.

D.3.2 Reducer Section

Using the procedure outlined in Paragraph D.3.1, prepare the reducer section to the dimensions shown in Figure D.1.

D.3.3 Anneal Test Capsules

Anneal test capsules (capsule and reducer section are separate units) in lots consisting of 250 units or less. Heat to 563°C (1040°F) and hold at this temperature for 0.25 hour. Heating rate is not critical. Cool from 563°C (1040°F) to ambient temperature at the normal furnace cooling rate.

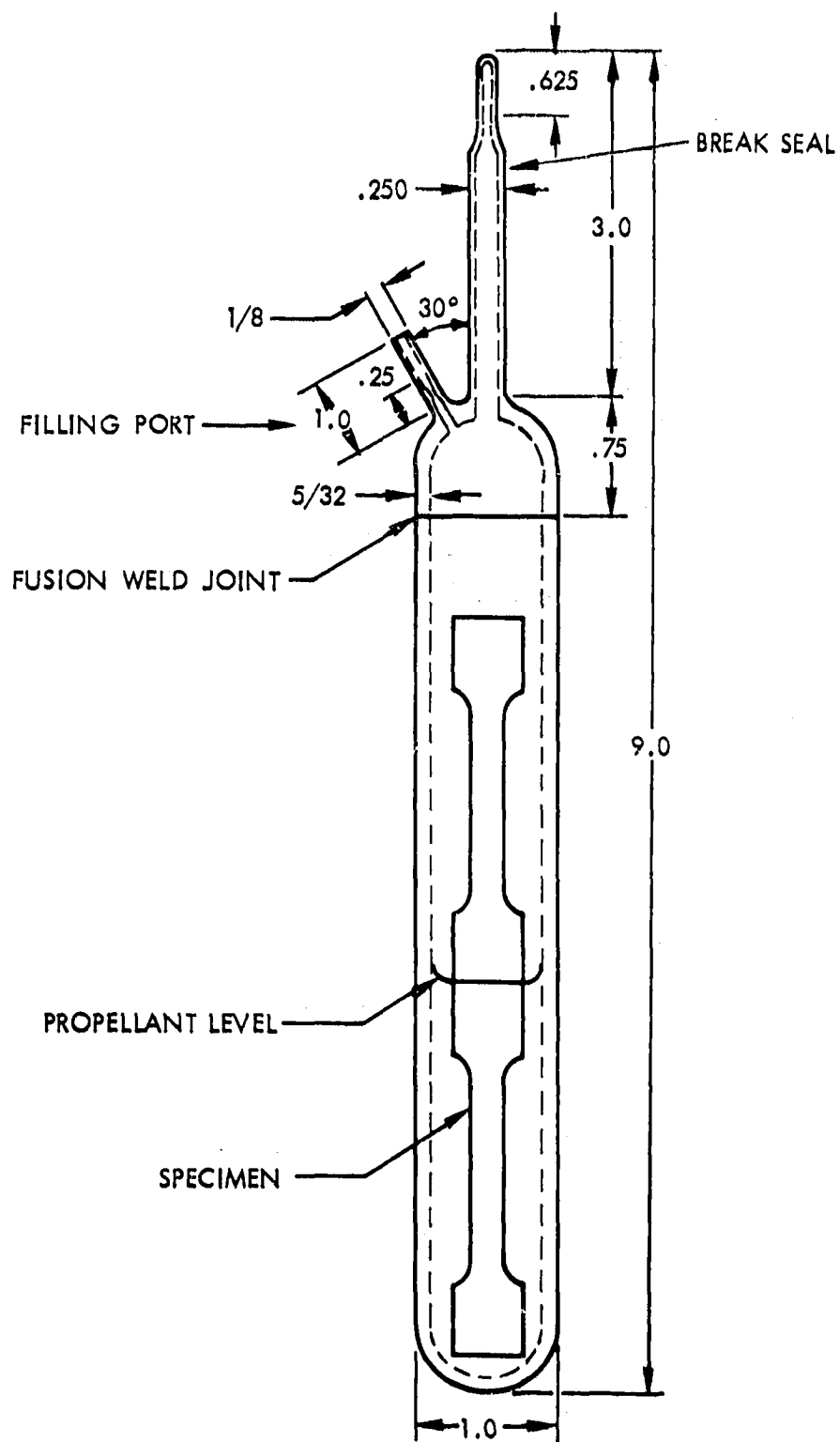


Figure D.1. Sealed Compatibility Test Capsule

D.3.4 Dissimilar Metal Specimens

For each set of dissimilar metal specimens, prepare from glass rods six "C" clamps that will hold the specimens snugly together.

D.3.5 Specimen Insertion

Clean the capsule and reducer sections, glass rods on "C" clamps in accordance with the instructions for titanium in JPL Specification FS506300, Revision A, Section 3.10.5 Newly Machined Parts.

Using white lint-free gloves to hold all external glass surfaces and with forceps having TEFLON coated tips, place a specimen in a capsule bottom. There are four different types of specimens: (1) Double "dog-bone", (2) welded or brazed double "dog-bone", (3) dissimilar metals, and (4) stressed. Place the double "dog-bone" and welded or brazed specimens into the specimen capsule bottom while using lint-free gloves to hold all external glass surfaces and TEFLON coated tipped forceps to place the specimens in the capsule bottom. Then place two glass rods, 0.3-inch in diameter and 2.5-inch long, on either side of the test specimen. Clamp the dissimilar metal specimens together with six glass "C" clamps and insert in the capsule bottom in the manner described above. Place the stress specimen and stress apparatus in the capsule bottom in the same manner as the other specimens, except do not add glass rods because there will not be sufficient room.

Fuse the reducer to the bottom of the test capsule as shown in Figure D.1. While fusing the reducer to the bottom, purge the capsule with dry filtered argon to avoid moisture condensation in the capsule. For the fusing operation use a hydrogen-oxygen flame. Constrict the 0.125-inch O.D. tube to be used as a filling port as an aid in completing the test capsule hermetical seal. Inspect the test capsule for glass strains with a polariscope. All strains from the reducer installation should be concentrated in the test capsule neck.

By means of the filling port tube, connect the capsule to a gauge and a source of oil-free filtered nitrogen. Pressurize the capsule to 60 psig. Capsules failing to pass this test must be reconstructed being careful to re-clean specimens. Bleed the nitrogen pressure down to one atmosphere and disconnect the capsule from the nitrogen source.

23162-6020-RU-00

By means of a rubber band, place a plastic bag over the filling port. Tag the capsule by placing over the break-seal tube a stainless steel tag scribed with the sample number. The capsule is now ready for transporting to the test site.

APPENDIX E

PROCEDURE FOR PROPELLANT FILLING OF CAPSULES

E.1 SCOPE

This procedure describes the propellant loading of specimen capsules, sealing of capsules, and the placing of capsules in storage canisters for transporting to -320°F conditioning flasks.

E.2 PURPOSE

The purpose of this procedure is to provide sufficient detail for the safe filling of propellant into specimen capsules.

E.3 FILLING CAPSULES

E.3.1 Preparing Capsules for Loading

Connect 0.125-inch stainless steel tubing to valves 1-5 shown on the Propellant Handling Manifold drawing (Figure E.1). By means of a Swagelok fitting with Teflon ferrules, connect four filling ports on four compatibility test capsules. By means of a vacuum pump attached at valve 14, with valves 1, 9, 13, 15, 16 and 19 closed, evacuate the manifold and specimen capsules for 10 minutes. With safety shield in place, close valve 14 and introduce approximately 1.1 atmospheres of propellant from propellant supply tank. (The following section describes the introduction and removal of the pre-treatment of passivation propellant.) Allow the propellant to remain in the system for 10 minutes and then evacuate the system.

E.3.2 Introduction and Removal of Pre-Treatment Propellant

By means of the line attached to valve 1, F_2 or FLOX is introduced to the system. Connect the supply tank to a fluorine pressure reducing regulator, designated as No. B-15F-F70M by the Matheson Company. Connect this valve to a rod extending through a barricade that is to be used for setting the regulator at 10 psig. Connect the F_2 or FLOX cylinder to a manually actuated gear box that can be operated by an extension handle

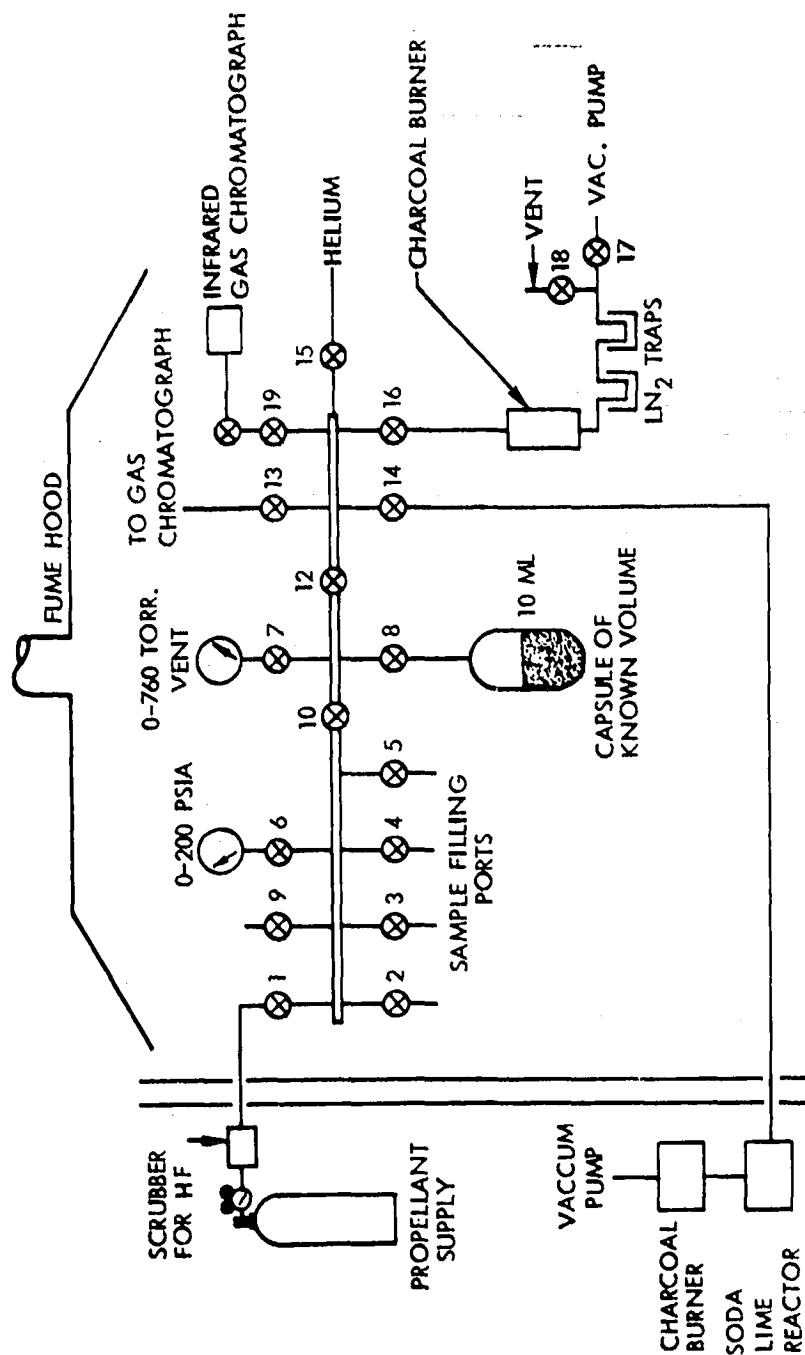


Figure E.1 Propellant Handling Manifold

passing through a barricade. Connect the gas regulator with 0.25-inch stainless steel or copper tubing to a sodium fluoride packed trap. Then by means of 0.25-inch stainless steel or copper tubing, connect the sodium fluoride trap to the manifold.

With valve 1 closed, open the F_2 or FLOX supply cylinder and adjust the regulator to 10 psig. Introduce by valve 1 about 1.1 atmosphere of F_2 or FLOX into the system. After 10 minutes, remove the propellant by means of a vacuum pump attached to valve 17 with valve 18 closed. Allow the propellant to pass over a charcoal burner by opening valve 16. Condense the CF_4 formed by the charcoal burner in liquid nitrogen cooled metal traps. At the end of each day, discharge through a vent tube into a hood or high vent line the CF_4 and possibly oxygen in the traps by closing valves 16 and 17, opening valve 18 and allowing the traps to warm.

E.3.3 Loading of Propellant into Capsules

Attached at valve 8 an empty glass sample capsule that has an external mark indicating the 10 ml volume required to fill the capsules containing the double dogbone specimens to a level between the two connected dogbones. Then open valve 14 and evacuate the manifold and sample capsules (valves 2, 3, 4, 5, 6, 7, 8; 12 and 14 are opened; valves 1, 9, 13, 15, 16 and 19 are closed). Close valves 2, 3, 4, 5 and 14. Then by means of valve 1, slowly allow propellant to enter the manifold and empty sample capsule that has been placed in a liquid nitrogen cooled Dewar. Do not allow the pressure in the manifold to exceed 30 psia. Place each of the sample capsules in a Dewar filled with liquid nitrogen to precool the sample capsule. Then place one capsule in liquid helium, open the valve to that sample capsule, and allow the propellant to transfer to the sample capsule from the known volume capsule containing the propellant.

By means of a four-foot extension on a hydrogen-oxygen torch, seal the filling port on the sample capsule. (This operation should be performed using all safety precautions because it is the most hazardous operation in the entire plan.)

Remove with tongs the first sample capsule from liquid helium and place in a canister (Figure E2) that is in a liquid nitrogen Dewar attached to a cart with a safety shield mounted. Repeat the filling operation for the other three connected sample capsules. Transfer the loaded canisters

23162-6020-RU-00

to the conditioning flasks. The above operation can be repeated until all the sample capsules are loaded.

E.4 PROPELLANT SAMPLING

Collect a gas phase sample of both propellants according to Appendix F, "Procedure for Analysis of Propellant Samples."

SAFETY NOTES: Anyone working with F_2 and FLOX should be familiar with all safety precautions recommended by the suppliers. Protective clothing, neoprene gloves and face shields should be worn at all times. Safety shields should be between the operator and all equipment containing the propellants.

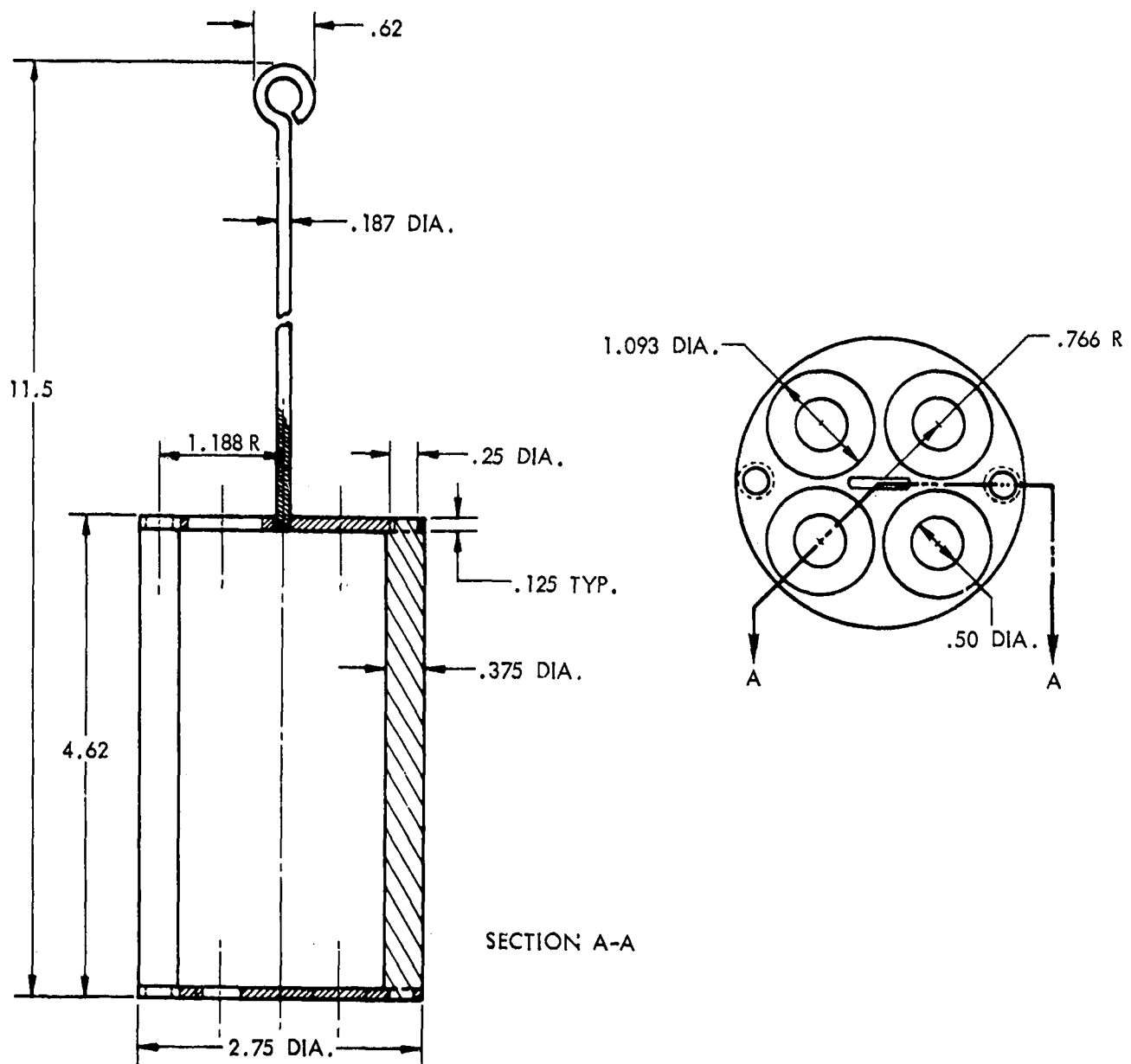


Figure E.2. Canister Modification

APPENDIX F
PROCEDURE FOR ANALYSIS OF PROPELLANT SAMPLES

F.1 SCOPE

This procedure describes the analysis of gaseous propellant samples.

F.2 PURPOSE

The purpose of this procedure is to provide details for the analysis of gaseous samples. Samples are to be taken of the initial propellant and the propellant removed at the completion of compatibility tests.

F.3 SAMPLE ANALYSIS

F.3.1 Initial Propellant Samples

Evacuate the propellant handling manifold through valve 16, with valves 1, 2, 3, 4, 5, 15 and 18 closed. (See Figure F1). Then fill the manifold with 240 torr of propellant in the manner described in Appendix E, "Procedure for Propellant Loading of Capsules," Section 2.3, Loading of Propellant into Capsules." Valve 13 is connected by means of a 0.125-inch stainless steel tube to a 1 cc stainless steel gas chromatograph gas sampling loop. Valve 19 is connected to an infrared gas sample cell. Then fill the gas chromatograph sample loops and infrared gas sample cell. Close the valve on the infrared gas cell and inject the propellant sample into the gas chromatograph for analysis according to the procedure described in F.3.2. Then evacuate the manifold by pumping through valve 16. Then through valve 18 add helium to a pressure of 790 torr. Close valve 18 and open the valve on the infrared gas cell for about two seconds. This brings the pressure in the gas cell to 760 torr. Analyze the contents of the infrared cell according to the procedure described in F.3.3. After filling the infrared gas cell close the valve on the sample bomb, and evacuate the manifold by pumping the propellant through valve 16 and the charcoal burner. The traps in Figure F.1 are liquid nitrogen cooled to collect CF_4 . At the end of each day, allow the trap to warm and vent the contents through valve 18.

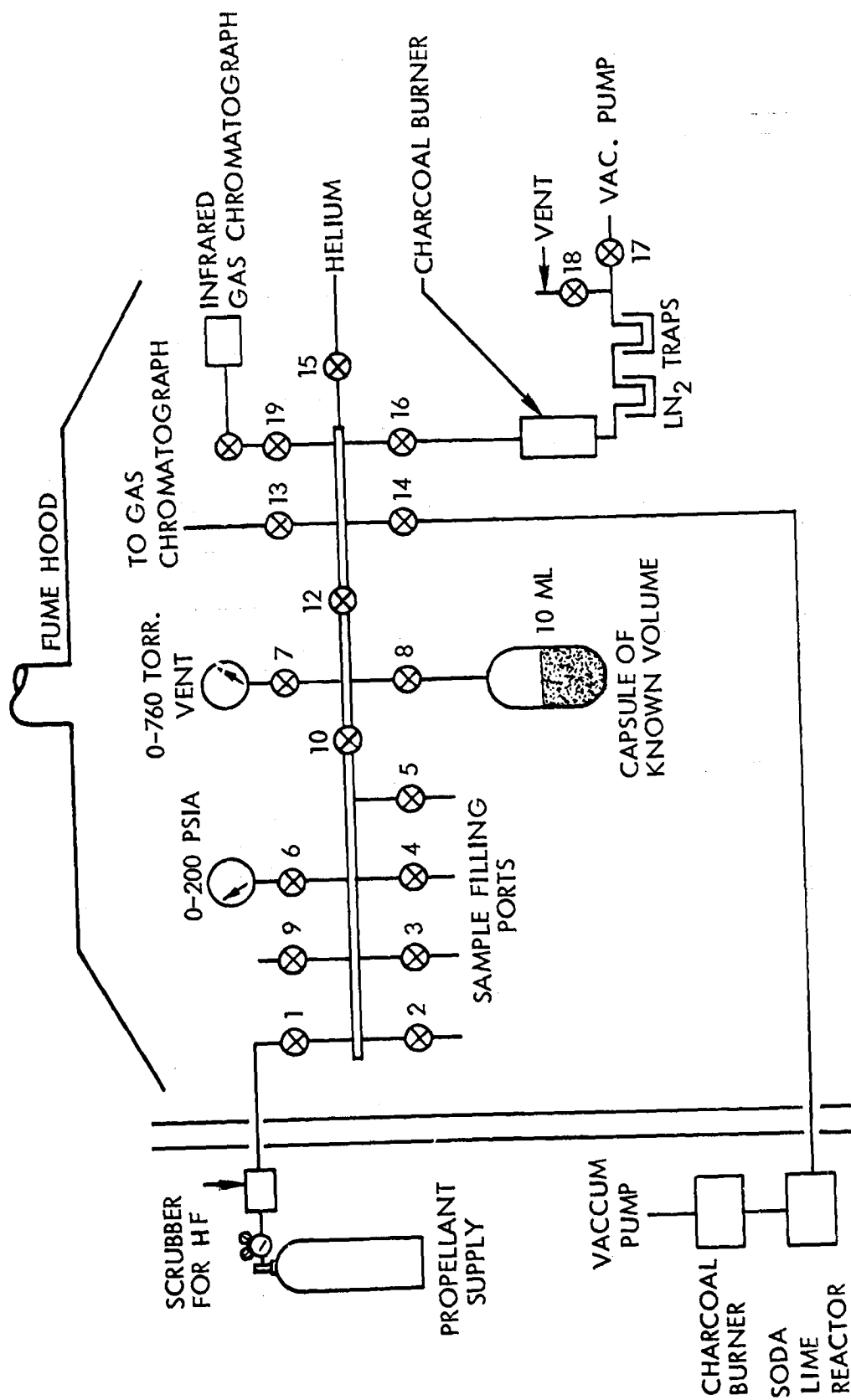


Figure F.1. Propellant Handling Manifold

F.3.2 Gas Chromatographic Analysis

As described in F.3.1 gas phase samples are introduced at the vapor pressure of the samples (about 280 torr) at -320°F into a 1 cc gas chromatograph sampling valve constructed of stainless steel. The analysis is performed from this point on according to the procedure in MIL-P-24713, "Specification, Propellant, Chlorine Pentafluoride," 2 May 1969 except that an 18-inch x 0.25-inch O.D. reactor filled with sodium chloride is placed before the Fluoropak 80 coated column. The sodium chloride reactor converts fluorine to chlorine which permits the separation of large quantities of oxygen and chlorine on the gas chromatograph column. The normalization factors for oxygen and fluorine (measured as chlorine) must be determined each day. Because fluorine, carbon dioxide, carbon tetrafluoride and silicon tetrafluoride are present in small quantities, these components are determined by infrared analysis.

F.3.3 Infrared Analysis

An infrared cell identical to that described in MIL-P-27413 is used except the cell length is seven inches. Standard curves for each of the substances listed below along with the absorption frequencies used are listed below.

<u>Substance</u>	<u>cm⁻¹</u>
HF	3770
CO ₂	2350
CF ₄	1280
SiF ₄	1050

All spectrum were run in the cell brought to a total pressure of 760 torr with helium. Samples withdrawn from sample capsules are run in the same manner as the standard curves and absorptions compared to those of the standard curves.

F.4 SAMPLING OF CAPSULES

At the completion of the compatibility testing, take three gas phase samples and analyze as described above. Take one sample when the sample capsule is opened (vapor pressure will provide a sample at 280 torr), another when about half the propellant has evaporated, and a third as the

last few drops of propellant are evacuated. The removal of propellant from specimen capsules is described in Appendix H, "Procedure for Removing Propellant from Specimen Capsules."

After sampling operations, evacuate and then fill the propellant handling manifold with slightly over 1 atmosphere of helium to prevent moisture from entering the manifold.

APPENDIX G

PROCEDURE FOR STORAGE OF SPECIMENS

G.1 SCOPE

This procedure describes the placing of canisters loaded with specimen capsules into the temperature conditioning flasks, the storage of specimens, and the removal of canisters from the temperature conditioning flasks.

G.2 PURPOSE

The purpose of this procedure is to provide sufficient information for the safe loading, storage, and unloading canisters from the temperature conditioning flasks and a description of how the controls are to be operated for delivering liquid nitrogen into the conditioning flasks.

G.3 SPECIMEN STORAGE

G.3.1 Loading of Canisters into Conditioning Flasks

Set the controller for the primary liquid nitrogen (LN_2) supply at -320°F (see Figure G.1). Set the controller for the emergency LN_2 supply at -300°F . Turn the controller on the primary LN_2 supply to -320°F and allow the conditioning flask to fill about half full of LN_2 . When the thermocouple attached to the controller continuously reads -320°F , the flask should be half full. Turn off the LN_2 supply and visually determine the level of LN_2 in the flask. Leave the primary controller set at -320°F . Connect the thermocouples as shown in Figure G.1.

Transport one canister loaded, as described in Appendix E, to the temperature conditioning flask. The canister is in LN_2 on a cart with a safety shield. Then place the canister into the conditioning flask, and hook the handle into the slot provided at the mouth of the flask. Repeat this procedure until the flask is loaded with nine canisters.

G.3.2 Storage of Specimen

Determine daily that LN_2 controllers are functioning properly and that the LN_2 supply tanks contain adequate supplies of LN_2 . If a malfunction develops, fill the storage flask with LN_2 by adjusting emergency

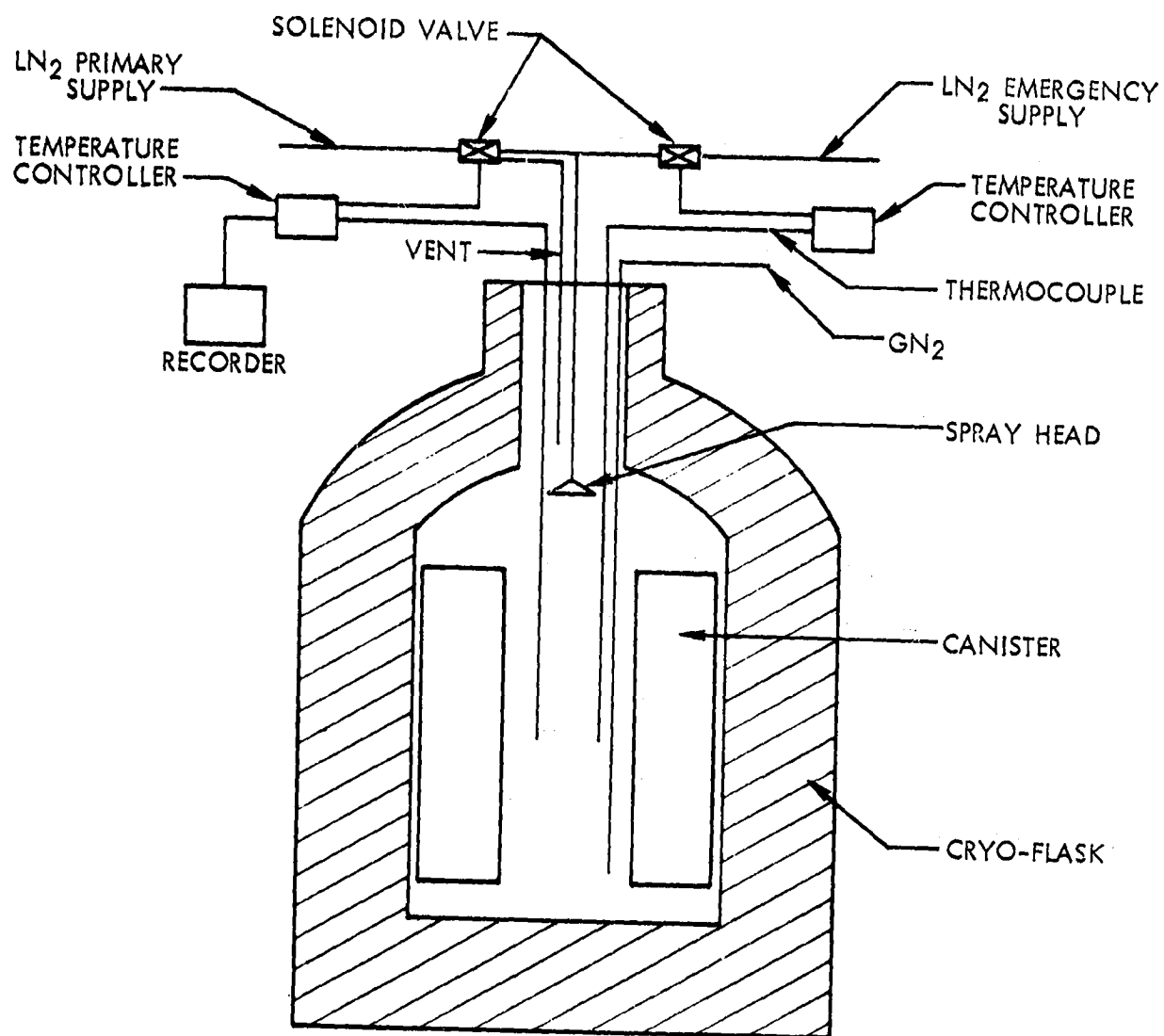


Figure G.1. Conditioning Flasks and Controls

controller to -320°F . If the electrical power service is interrupted, the normally open solenoid valve on the emergency LN_2 supply line will open and fill the flask with LN_2 .

G.3.3 Unloading of Canisters from Temperature Conditioning Flasks

Care must be exercised when unloading the temperature conditioning flasks because the pressure in the specimen containers is unknown. Remove one canister at a time from the conditioning flask and place the canister in a LN_2 cooled Dewar mounted to a cart equipped with safety shields. Push the cart to the test cell for opening the specimen capsules.

APPENDIX H

PROCEDURE FOR REMOVING PROPELLANT FROM SPECIMEN CAPSULES

H.1 SCOPE

This procedure describes the removal of propellant from the specimen capsule.

H.2 PURPOSE

The purpose of this procedure is to describe the safe unloading of propellant from the specimen capsule.

H.3 OPENING CAPSULE

H.3.1 Connecting Capsule to Propellant Handling Manifold

While the canister is in the LN₂ filled Dewar, raise the bottom plate of the canister (Figure H.1) by lifting with tongs the 0.125-inch rod attached to the bottom plate of the canister. Remove with tongs the metal protective tube from one of the capsules and by means of tongs remove the specimen capsule from the canister. Immediately place the specimen capsule in a LN₂ filled Dewar flask. While immersed in LN₂, manipulate the specimen capsule into position for connection to the opening device (Figure H.2). The opening device located in a test cell is connected by a 0.125-inch line to Valve 2 (Figure H.3). Connect the specimen capsule to the opening device using a Swagelok nut and Teflon ferrules as indicated in Figure H.2. While the manifold is evacuated, open valve 2 (Figure H.3) and allow the tip of the specimen capsule to remain in a vacuum for 15 minutes to remove any moisture on the surface of the capsule tip.

H.3.2 Opening of Specimen Capsule

With valves 1, 3, 4, 5 and 12 closed, and valves 13 and 19 opened, (Figure H.3), use a 28-volt power supply to actuate the solenoid of the opening device which will crush the tip of the specimen capsule. Observe if the pressure in the system increases by observing the 0-760 torr gauge on the propellant handling manifold. Note the pressure on the Test Specimen Data Sheet for the specimen in the capsule.

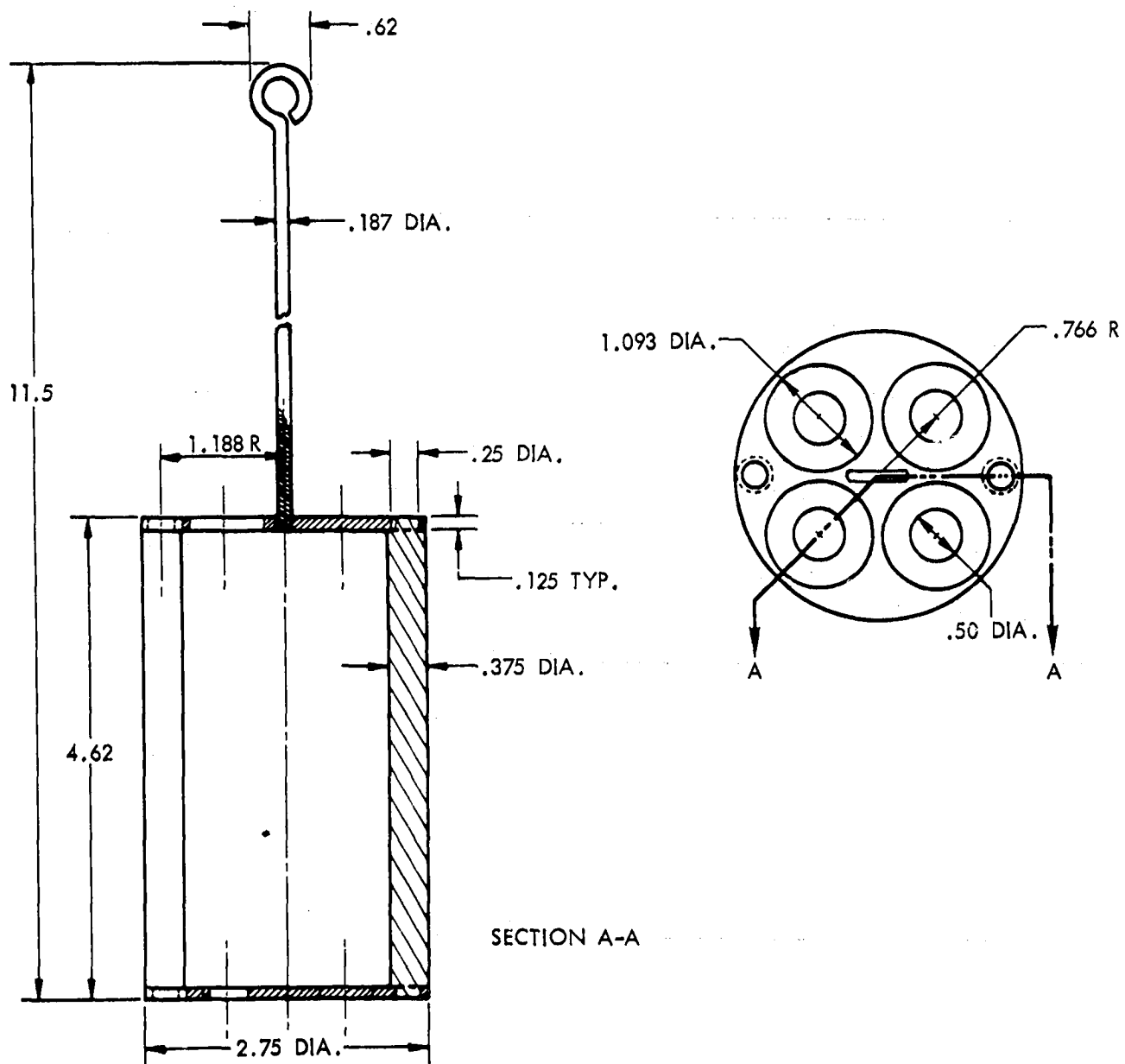


Figure H.1. Canister Modification

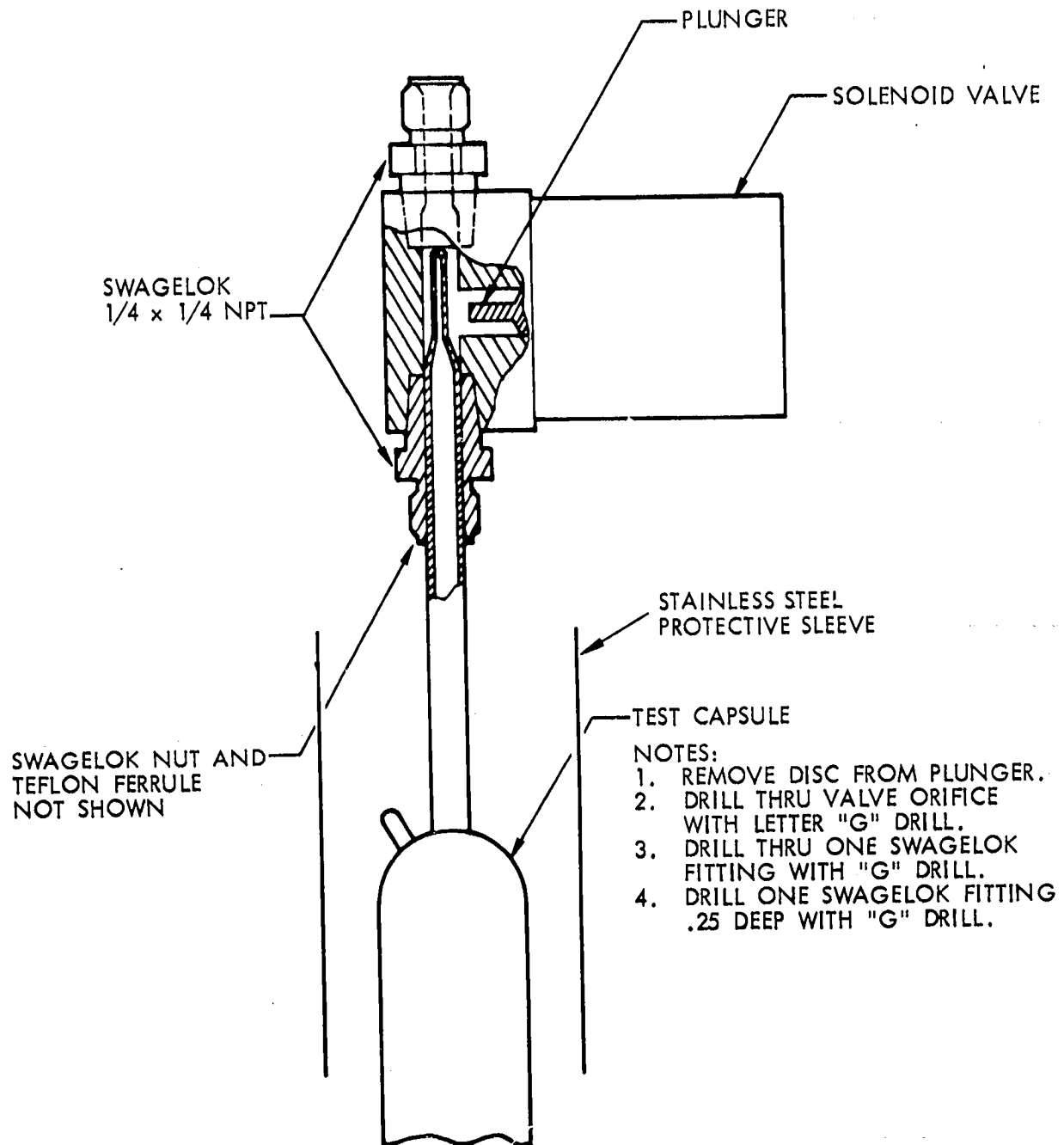


Figure H.2. Capsule Opening Device

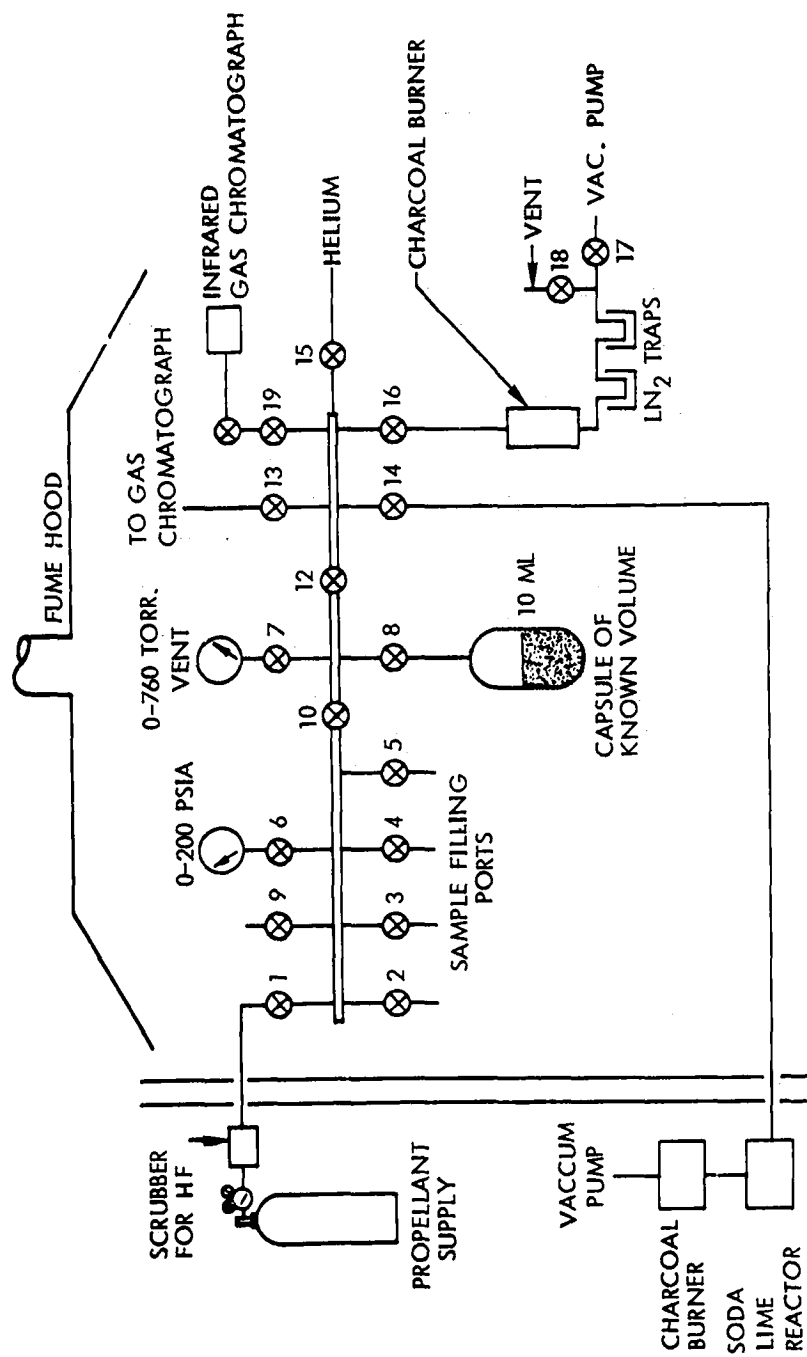


Figure H.3. Propellant Handling Material

Obtain an infrared spectrophotometric and a gas chromatographic sample of the gas phase according to the procedure described in Appendix F. Close valve 2, attach the infrared gas cell at valve 19, open the valve on the infrared gas cell, and open valves 13 and 19. Evacuate the manifold through valve 16. Close valves 13, 16 and 19 and open valve 2. Open valve 14 and vacuum distill the propellant from the capsule. The propellant is passed through a soda lime and then a charcoal scrubber before entering the vacuum pump. The temperature of the soda lime reactor and charcoal burner is measured by attaching a thermocouple to each with tape. When the soda lime scrubber reaches 130°F or the charcoal scrubber temperature begins to increase, the distillation is stopped until the reactors cool (110°F for the soda lime scrubber). When about half of the propellant has been removed from the capsule, gas chromatographic and infrared spectrophotometric analyses are performed as described above. Again, when only a few drops of propellant remain in the capsule, gas chromatographic and infrared spectrophotometric analyses are performed.

H.3.3 Closing of Specimen Capsules

By means of a hydrogen-oxygen torch seal the tip of the specimen capsule. The capsule is filled with 1.0 atmosphere of helium at this time. By ordinary handling techniques, the specimen capsules are shipped to a metallurgical laboratory for examination.

APPENDIX I

PROCEDURE FOR POST TEST EXAMINATION

I.1 SCOPE

This procedure describes the post test examination of propellant and specimens.

I.2 PURPOSE

The purpose of this procedure is to describe the techniques that are recommended for post test examination of propellant and specimens.

I.3 EXAMINATION OF PROPELLANTS

The propellant samples shall be analyzed according to the procedure described in Appendix F, Section F.3.1 Initial Propellant Samples. The analytical results for FLOX will not be identical with those obtained on the initial supply, but the results will have to be compared with control samples. The reason for this is that fluorine and oxygen have different vapor pressures at -320°F.

I.4 EXAMINATION OF TEST SPECIMENS

Pack in a box to protect from breakage. Capsules sealed according to the procedure in Appendix H, Section H.3.5. Transport the sealed capsules to a clean room for opening. By means of diamond tool scratch a line around the capsule and open by placing a heated rod against the scratch on the capsule. The final opening operation may be performed in a dry box if this appears desirable. Collect any residue in the bottom of the capsule, weigh residue and then submit it to identification analysis. (The method of analysis must be selected at this time, but x-ray diffraction is recommended.) Weigh the specimen and carefully remove any adhering salts with a Teflon coated tool. Weigh the adhering salts and submit them to the same analysis as the residue found in the capsule. Examine the specimen by means of a microscope. If any changes are noted at the propellant-vapor interface line, cut this section from the specimen for metallurgical examination. If no changes are noted at the interface line, cut the two dog-bones apart and submit both specimens to tensile testing in accordance with ASTM Method E8-57T. Record all results on the forms

23162-6020-RU-00

shown in Figures 16-18 of the final report. If corrosion is detected by visual and microscope inspection or by tensile testing, perform a detailed metallographic examination on corroded materials.